

**FABRICATION AND TESTING OF IMPROVED CELLULOSE  
ACETATE MEMBRANES IN TUBULAR FORM FOR  
LOW PRESSURE REVERSE OSMOSIS**

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In Partial Fulfilment of the Requirements  
for the Degree of**

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**By**

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**to the**

**DEPARTMENT OF CHEMICAL ENGINEERING**

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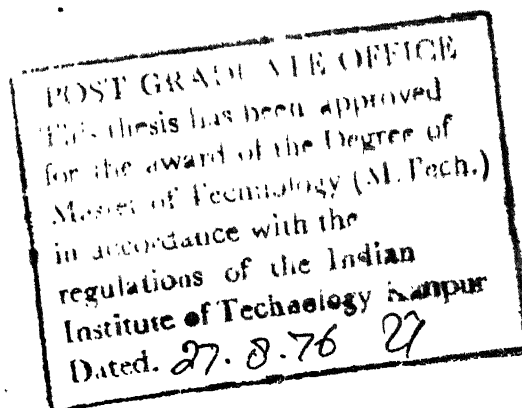
CERTIFICATE

This is to certify that the present work 'FABRICATION AND TESTING OF IMPROVED CA MEMBRANES IN TUBULAR FORM FOR LOW PRESSURE REVERSE OSMOSIS' has been carried out under my supervision and has not been submitted elsewhere for a degree.

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[M. Murali Krishna]



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ABSTRACT

An apparatus has been constructed which enables the preparation of assymetric cellulose-acetate membranes in tubular forms by the 'Casting Leaching' method. Adequate control of physical parameters which affect the structure and properties of these membranes have been developed. Two tubular test cells of different design have been fabricated and tubular membrane heat treatment has been successfully carried out without end leakages. Membranes cast with composition near the phase boundary and with low evaporation periods have been tested with a 5000 ppm sodium chloride feed solution at 250 psig with and without annulus inside the membrane tube for low feed flow rates in the laminar region. Different designs of turbulent promoters have been used on the annulus in the feed flow. Considerable improvement in flux and rejection of the membrane results from the use of annulus and turbulent promoters in the test cell. The membrane tubes tested in flat form perform as well as the best flat membranes prepared. The tubular membrane performances are lower than the performances of the flat membranes due to severe concentration polarization resulting from a low Reynolds number laminar flow in the test cell.

## CHAPTER 1

### INTRODUCTION

Reverse osmosis (also known as hyperfiltration) classified as a 'pressure-activated membrane moderated, isotherm operation' [1] is a relative newcomer to the repertory of separation techniques available to a chemical engineer for separation of liquid mixtures and solutions. In principle, the process is useful for separation, concentration, and fractionation of small molecular weight organic or inorganic substances in aqueous or nonaqueous solutions in the liquid phase. Hence it opens up a new and versatile field of separation technology in chemical process engineering. RO (Reverse-Osmosis) process has been applied to various industrial separation problems:

(a) desalination of brine water: normal operating pressures in RO plants for such processes vary depending on the feed concentration (400-600 psig for 0.5 per cent salt solution, 600-800 psig for 1.0 per cent salt solution and 1000-1500 psig for 3.5 per cent salt solution); brackish water contains 1500-7000 ppm and sea water contains 35000 ppm of salt; [2]

(b) pollution control: (1) The waste effluents from metal finishing plants contain numerous toxic constituents

e.g., copper, lead, zinc and chromium. The waste constituents also have considerable economic value. Using RO separation technique, product water of almost any desired quality can be produced by repeated operations. (2) Waste water from the paper and pulp industry: An average size pulp mill producing 150-500 million tons of cellulose pulp may commonly consume 5-50 million gallons water daily. A large part of this water can be reused after processing by RO. The concentrated waste may then be disposed of economically or the chemicals in it can be recovered.[3].

(c) Separation and concentration of viral and bacterial suspensions: RO membranes have the ability to separate viruses and therefore RO is being increasingly used in the Pharmaceutical industry and in municipal sewage treatments.

(d) Concentration of liquid foods and perfumes: liquid food in natural state contains 80-90 per cent water which adds volume and weight, thus making the transportation and storage more expensive [4]. Orange juice, apple juice, milk, coffee, tomato juice cheese-whey and egg white are some food products where RO method of water elimination is finding increasing use. In many cases, liquid foods are sensitive to high temperatures which reduce their flavor and nutritional value in the other separation process commonly used e.g. evaporation. In the case of perfumes also, flavor value can be saved by using RO [4].

(e) Concentration of sugar solutions: Huge quantities of

sugar can be obtained from sugar cane waste bagasse by washing with water and concentrating by RO technique. Waste sugar cane cake washing gives roughly 0.1 per cent solution which can be economically concentrated by RO to atleast 20 per cent solution without much difficulty [3],[4].

(f) Fractionation of multicomponent solutions: Based on differences in permeabilities of various components through membrane, separation can be effected. For example, spent solut from soap industry contain some salt beside glycerine. Glyceri can be separated by using a suitable membrane which allows onl glycerine to pass through and rejects salts [5].

(g) Concentration of dye-waste has also been carried out with RO.

In recent years, extensive work has been done and considerable progress has been made in RO technology. Various membrane configurations that can be used are flat, tubular, spiral wound and hollow fibre [2]. The various criteria for selection amongst these membrane forms and their corresponding modules are as follows: the flow paths in the module should provide low resistance to the flow over membrane; the module should be simple in design and fabrication, have a high surface to volume ratio and should have sufficiently good performance characteristics; lastly the membrane form should be produced easily. Spiral wound and hollow fibre modules have very good surface to volume ratio but the problems involved in casting and mounting of such membranes are not inconsiderable [2]. Tubular membranes. besides providing low

resistance flow paths have several inherent advantages [6].

(a) the membrane support tube combines the function of a pressure vessel and membrane support surface thus effecting a design simplification; (b) the flow of fluid is ensured past every square centimeter of membrane surface area without the necessity of installing baffles. For this reason, particulate matter deposition problems are lessened, feed pretreatment problems are simplified and chemical or hydraulic cleaning of membranes is more certain; (c) by means of appropriate valving the tubular membrane design lends itself to ready replacement of tube units without the necessity of shutting down the entire plant. Because of the above advantages over other forms of membrane, the tubular membranes find extensive commercial use and precise characteristic equations for concentration polarization in tubular membranes are also available in literature [7].

RO membranes can be made with various polymers such as cellulose-acetate, nylons, polyelectrolytes. But CA (cellulose acetate) is overwhelmingly favored as the membrane since it is highly productive and the technology is known. The various criteria for a good membrane material [8] are that (1) it must be highly permeable to water and impermeable to solutes; (2) the membrane barrier must be as thin as possible consistent with strength requirements; (3) the membrane should be chemically inert, mechanically strong and creep resistant;



and (4) the membrane should be capable of being fabricated into configurations of high surface to volume ratio. Asymmetric CA membranes of Loeb-Sourirajan type [96] satisfy all the above criteria.

The earliest tubular membrane casting technique using the gravity drop of the casting tube around a stationary bob was developed by Loeb [6]. Other investigators who have also developed tubular membrane casting techniques are Sourirajan et al. [10], Richardson et al. [11] and McCutchan and Johnson [17]. While Richardson et al. [11] developed a technique for the continuous and automatic production of tubular membrane around a stationary casting bob, Sourirajan et al. [10] have devised an arrangement for moving the casting bob inside a stationary casting tube. In the present work, Loeb's [6] simple gravity drop technique was adopted with modifications to control the speed of fall of the casting tube around a stationary bob. Further McCutchan and Johnson's [12] recommendations regarding the materials of construction and McKinney's [13] casting bob design with a top guide arrangement have been incorporated to produce tubular membranes.

An extensive amount of international research effort has been directed towards improving the CA membrane over the original Loeb-Sourirajan membrane. These efforts have been summarized in a recent thesis by Pandurangaiah [14] who following up the preliminary work of Agarwal [15] has developed

highly productive CA membranes from casting compositions and conditions not encountered in literature. The new flat CA membranes produced by Pandurangaiah [14] are sufficiently productive when compared with existing membranes to merit immediate commercial exploitations. In fact, Pandurangaiah [14] has shown that his membranes can effectively desalt 5000 ppm brackish water at a very low pressure of 250 psig ( $17.6 \text{ Kg/cm}^2$ ) when commercially 400-600 psig are ( $28.18$  to  $42.27 \text{ Kg/cm}^2$ ) are used. Such low pressure operation is expected to result in substantial savings in the pump costs as well as in the operating costs. Further, it is known that with time, membrane water flux decreases slowly due to the phenomenon of membrane compaction resulting from an irreversible creep compaction of the pore structure in the membrane skin. It is also known, that the higher the pressure of operation, the greater is the membrane compaction and the larger is the long term flux loss [16]. At 250 psig operation ( $17.6 \text{ Kg/cm}^2$ ), such flux loss is expected to be minimum if not negligible.

Experiments conducted by Pandurangaiah [14] spanned an air evaporation period of between 0.05 seconds to 12 seconds. The flat membranes of Pandurangaiah [14] performed identically in an air evaporation period ranging between 0.05 to 0.5 seconds. Therefore it is obvious that there is sufficient evaporation period available to make improved tubular. CA membranes as long as the speed of casting can be maintained at the proper

level which will allow sufficient gap between the water level and the casting bob to control the atmosphere in the gap. This atmosphere may have to be kept supplied with air so that acetone concentration does not reach too high a value inhibiting the skin formation.

A major obstacles which often limits the performance of reverse-osmosis membranes is the phenomenon of concentration polarization. The term concentration polarization [7] is given to the build up of solute layer, adjacent to the membrane surface which is more concentrated than the feed solution. Concentration polarization is an inherent consequence of the membrane's affinity for transmitting water in preference to the solute and is a general phenomenon which occurs to some extent in all separation processes. Concentration polarization has several deleterious effects on reverse osmosis; it increases the osmotic pressure at the membrane/brine interface and thereby decreases the effective driving force for water flow through the membrane since water flux is given by  $N_{\text{water}} = A(P - \pi)$  where A is a constant for the membrane, P is the applied pressure difference and  $\pi$  is the osmotic pressure difference between the feed solution and the production solution; it increases the driving force for the transmembrane solute flux and hence, it increases the salinity of the product water; it can lead to membrane fouling due to precipitation or gelation of sparingly soluble solutes on the membrane surface

and it may accelerate chemical deterioration of the membrane owing to the higher concentration of aggressive species at the membrane solution interface. A proper study of concentration polarization at the tubular membrane surface could not be carried out in this study because of the lack of a pump with sufficiently high capacity. Since the pump available has a pretty low capacity of only 0.8 litres/min, some annular arrangements were made to investigate their possible usefulness in reducing concentration polarization in the tubular membranes. The necessity for such arrangements are obvious since while a Reynolds number of 10000 - 20000 is necessary [1] for attaining the fullest membrane potential (least concentration polarization) in a tubular membrane, the available Reynolds number in the present system was only around 500 - 1000.

The objectives of this work are:

(1) To design and fabricate an apparatus for controlled casting, formation and annealing of tubular CA membranes;

(2) To test tubular CA membranes for reverse osmosis desalination of a 0.5 weight per cent NaCl solution at an operating pressure of 250 psig;

(3) To test flat CA membranes obtained by cutting tubular membranes under conditions used in objective (2).

Gupta and Adhikari [17] made a preliminary arrangement for casting a tubular membrane. They were able to make a very crude membrane. In this work, we have altered their

set-up substantially and have cast extremely productive CA tubular membranes under controlled conditions. These membranes may be operated at 250 psig with 0.5 weight per cent brine solution for desalination.

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## CHAPTER 2

### TUBULAR MEMBRANE FABRICATION AND TESTING: EQUIPMENT DETAILS AND EXPERIMENTAL PROCEDURE

In this chapter, detailed descriptions are given of the methods by which cellulose acetate tubular membranes were produced, mounted in test cells, annealed at temperatures between 70-90°C with hot water and tested at various pressures for reverse osmosis desalination of brackish waters. This chapter is divided therefore into four sections: Section A deals with the procedure needed for casting a tubular membrane as well as the necessary equipment details. Section B describes the tubular membrane test cell and the procedure for mounting a tubular membrane inside the cell. In section C, the various conditions to be met and the procedure to be followed for annealing the tubular membrane in hot water are given. A brief description of the membrane testing in a high pressure flow loop is available in Section D.

Before dealing with the procedure for casting tubular membrane as given in section A, it is pertinent to give the necessary details about the chemicals required and the mixing conditions needed to produce the solution of cellulose acetate in acetone and formamide (and dioxane).

## CHEMICALS:

CA from the same lot (No. AC-2109) of Eastman E-398-3 powder (renamed as CA-398-3) was used in the as received state in all experiments since McCutchan and Johnson [12] found that drying CA has no effect on membrane performance. The chemicals: acetone, dioxane, formamide and magnesium-perchlorate were of analytical reagent quality. Laboratory grade sodium chloride and distilled water were used for preparing 0.5 per cent brackish water feed solution. Analytical grade sodium chloride was used for standardization of  $\text{AgNO}_3$  solutions used for the analysis of sodium chloride in the permeate and the feed [14].

## CASTING SOLUTION PREPARATION:

The casting solution was prepared in 170 gm. batches in a 250 ml. conical flask fitted with standard B-24 glass joint, and adapter. Liquid chemicals were weighed in 'weighing bottles' of 25 ml. volume and the polymer CA was weighed on a clean Mylar sheet. All weighings were carried out in a Mettler balance. The conical flask with the solution was then rotated by means of a Type TI-147(b) Tempo Rotating Film Vacuum Evaporator drive for about fifty hours. The temperature of the bath in which the flask was kept immersed partially, was maintained at around  $45^\circ\text{C}$  by regulated heat input. All the solution compositions reported in this thesis are in weight per cent [14].

#### A. HOW TO CAST A TUBULAR MEMBRANE?

Tubular phase inversion membranes can be cast either in continuous manner or batch manner. In the continuous process (Richardson et al. [11]), a tubular membrane is made by continuously squeezing the polymer solution in between a moving teflon cylindrical tape and a rigidly fixed circular casting bob. Each of the two endless flat moving teflon tapes form an annulus through which casting solution is extruded. The tapes take on the semicircular shape due to the presence of a guide shoe. The batch process consists of a casting bob hanging freely inside a casting tube with polymer solution over the bob. A thin continuous coating of polymer solution over the inside wall of the casting tube can be made either by pulling the bob upwards by using a constant speed motor (Sourirajan et al. [10]) or by allowing the casting tube to fall down by its own weight. The latter is called the gravity drop technique. This technique was chosen for this work because of its simplicity and low cost. The fall of the casting tube can be either free or controlled. In the present case the casting equipment was designed to control the falling speed of the casting tube. A uniform speed of fall around particular values is an important factor in getting a uniform performance all along the length of the membrane. The casting tube with a coating of membrane casting solution is allowed to fall into an ice cold gelation bath with some air gap in between.

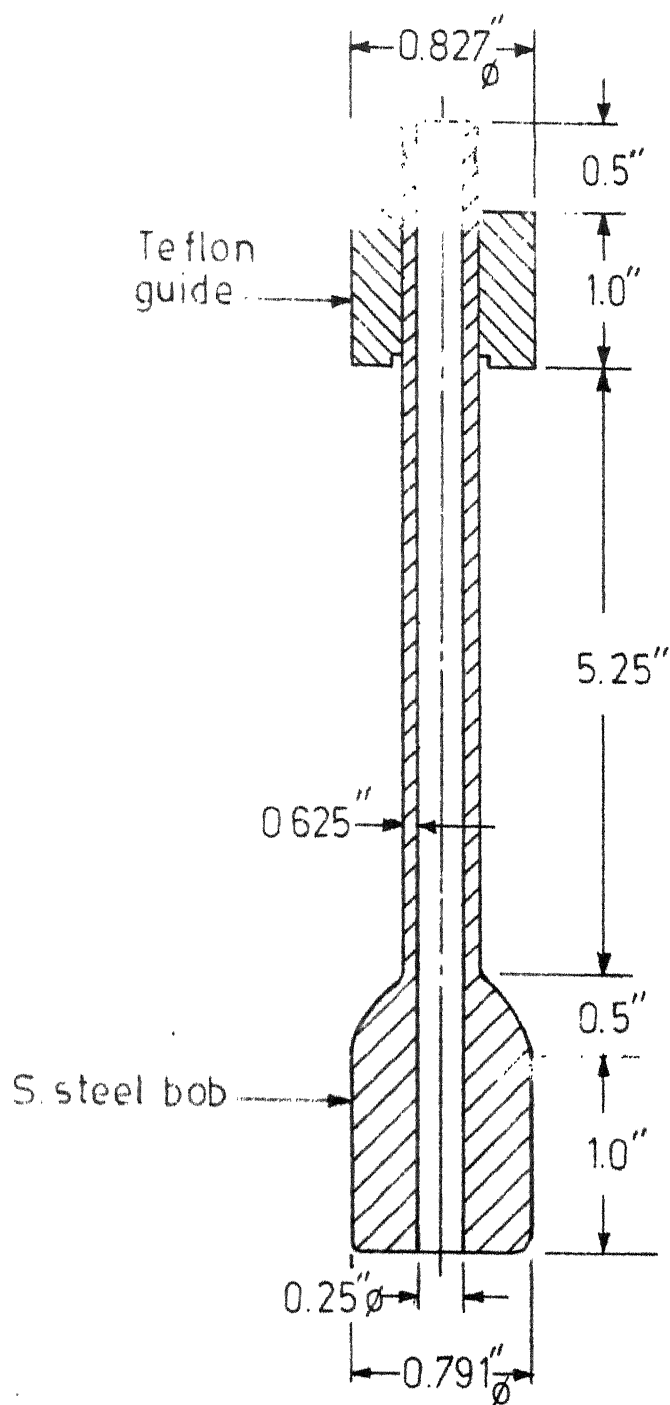


The gelation bath contains ice tap water mixture at  $0-1^{\circ}\text{C}$ . Gelation of the membrane takes place in this water bath with the leaching of formamide and acetone from the film. Gelation time required is roughly forty five minutes to 1 hour. The final membrane consists of an assymetric structure because of skin formation on the inside surface due to evaporation of acetone from casting solution during the travel of the membrane through the small air gap. Casting speed and the air gap height between the water level and the point of film formation control the acetone evaporation time. The membranes that have been cast with such an equipment are 46 inch long 0.800 inch I.D. and 0.816 inch O.D. which implies a thickness of eight thousandths of an inch. The membrane dimensions stated here are average values. There will be variation around the circumference as well as along the length. The casting tube length is 46 inch and usually two to six inches of the membrane at the starting end are not good because of initial effects. A detailed description of the various parts of the casting equipment follows:

(a) Casting Tube: The casting tube should have a high surface finish on the inside surface (less than 15 micro inches rms), dimensional accuracy (+ 0.002 inch, -0.001 inch) and high corrosion resistance. Corrosion resistance is the chief property looked for in the material of construction since it is essential that the tube maintain its dimensional accuracy

and surface finish with time. Brass tubes are somewhat unsuitable because formamide in the casting solution would cause corrosion which would necessitate frequent discarding of these tubes. Amongst the common materials available in tubular form, only glass and stainless steel are suitable. The glass blowing section of I.I.T. K. made a sample tube on order but it was found to be inaccurate dimensionally and has gross surface defects (Gupta and Adhikari [17]). Hence it was decided to go in for a stainless steel tube inspite of its high cost and machining difficulties. Ready made SS tubes lack both dimensional accuracy and the necessary surface finish. So, Small Arms Factory, Kanpur was approached for machining a 18/8 (18 per cent Cr, 8 per cent Ni) stainless steel tube of 46 inch long, 0.75 inch I.D. and 1.00 inch O.D. to 0.863 inch I.D. (This diameter was required to fit the membrane properly into the then existing 7/8 inch I.D. membrane test cell). A tube of 46 inch length 1 inch O.D. and 0.827 inch I.D. (due to machining problems I.D. could not be brought to our requirement) with desired surface finish was delivered, finally.

(b) Casting Bob: The corrosion resistance was the main criterion for the selection of material for the casting bob. Stainless steel 316 was chosen because of its corrosion resistance and easy machinability. Design requirements of the bob shown in the Figure 2. are (a) suitable shape for smooth



Material: S. steel  
and  
Teflon.

Fig. 2 - Tubular membrane casting bob.

flow of the casting solution into the annulus between the bob head at the bottom and the casting tube and (b) a piston type arrangement at the top of the bob to prevent acetone evaporation into the casting tube atmosphere. The piston type arrangement at the top, of the bob [13] also provides guidance to the falling casting tube to reduce the bad effects of any slight bend in the casting tube. Teflon was chosen for the piston material because it provides least friction against the casting tube without the use of any lubricant. It is also necessary to provide an arrangement for air circulation just below the bob and above the water level, as well as for water circulation inside the casting tube during the gelation period. This can be done by drilling 1/4 inch hole all the length of the bob at the centre and passing two copper tubes of 1/8 inch O.D. (arrangement of these tubes is explained later).

In our first attempt a casting bob was made which would leave a radial gap of 0.010 inch between the bob O.D. and the casting tube I.D. This would mean that bottom bob head is of 0.807 inch O.D. as the casting tube I.D. is 0.827 inch. To store the required amount of the casting solution (approximately 25 cc). The neck of 3/8 inch O.D. has to be about 5 inch long. Reducing the neck diameter would mean that the length of the casting bob could be reduced since a 1/4 inch hole has to be drilled through the bob, the smallest neck possible is of 3/8 inch O.D. The casting bob is supported by means of a string tied

to the circular grooves at the top of the bob. Adjusting the distance between the casting bob and the water level in the leaching tank allows one to vary the air exposure time for a given falling velocity of the casting tube. The cellulose-acetate membranes cast with this bob, resulted in membranes of 4-5 thousandths of an inch thickness. These membranes tubes were found to be difficult to handle in subsequent wrapping operations because of its flimsiness and it was decided to make another bob which could allow 0.018 inch as as-cast thickness (This gap was used by Sidney Loeb [6] without any difficulties in handling the cast membranes). To get a 0.018 inch as-cast thickness the bob bottom O.D. would have to be reduced to 0.791 inch diameter. To store the required quantity (35 cc) of polymer solution for extruding this thicker membranes the neck length would have to be 5.25 inch long. The membrane thickness obtained by using this bob varied between 0.007 inch - 0.010 inch. No handling difficulties were encountered with these membranes in the subsequent operations except often some membranes are formed with thinner sections along some lengths. These portions had to be rejected. The thinner sections are likely to have been formed due to relative deviations from the centre of the bob-casting tube combination. Perfectly centred bob in the casting tube yielded uniform membranes in a special experiment.

(c) Procedure for Solution Filling and Casting: For introducing polymer solution around the long neck of the bob a brass attachment was made since dimensional accuracy was not critical and corrosion can be tolerated. A brass tube of 1 inch O.D.; 7/8 inch I.D. and length 9.25 inch was threaded at one end to attach this to the casting tube using a socket as shown in the Figure 1. Three equidistant holes along the circumference were drilled one inch below the top of this brass filling tube. The diameter of the holes were chosen to fit a 50 cc polyethylene or nylon syringe end exactly. The holes were always closed with cello tape except during the injection of polymer solution into the filling tube to prevent acetone loss. Before filling began, the filling tube was positioned to cover the bob completely and to bring the three holes just below the teflon piston of the bob. Then equal quantities of polymer solution was injected into the filling tube through each hole using the syringe. The system was then kept stationary for one or two minutes to allow the polymer solution to distribute itself uniformly over the bob. It was found that 35 cc solution was just sufficient to give the full length of the membrane i.e. 46 inch after allowing for necessary wastage in the solution. After filling is complete, casting tube extension assembly with the filling tube was lowered a little so that the bob passes from the filling tube to the casting tube. The extension tube was

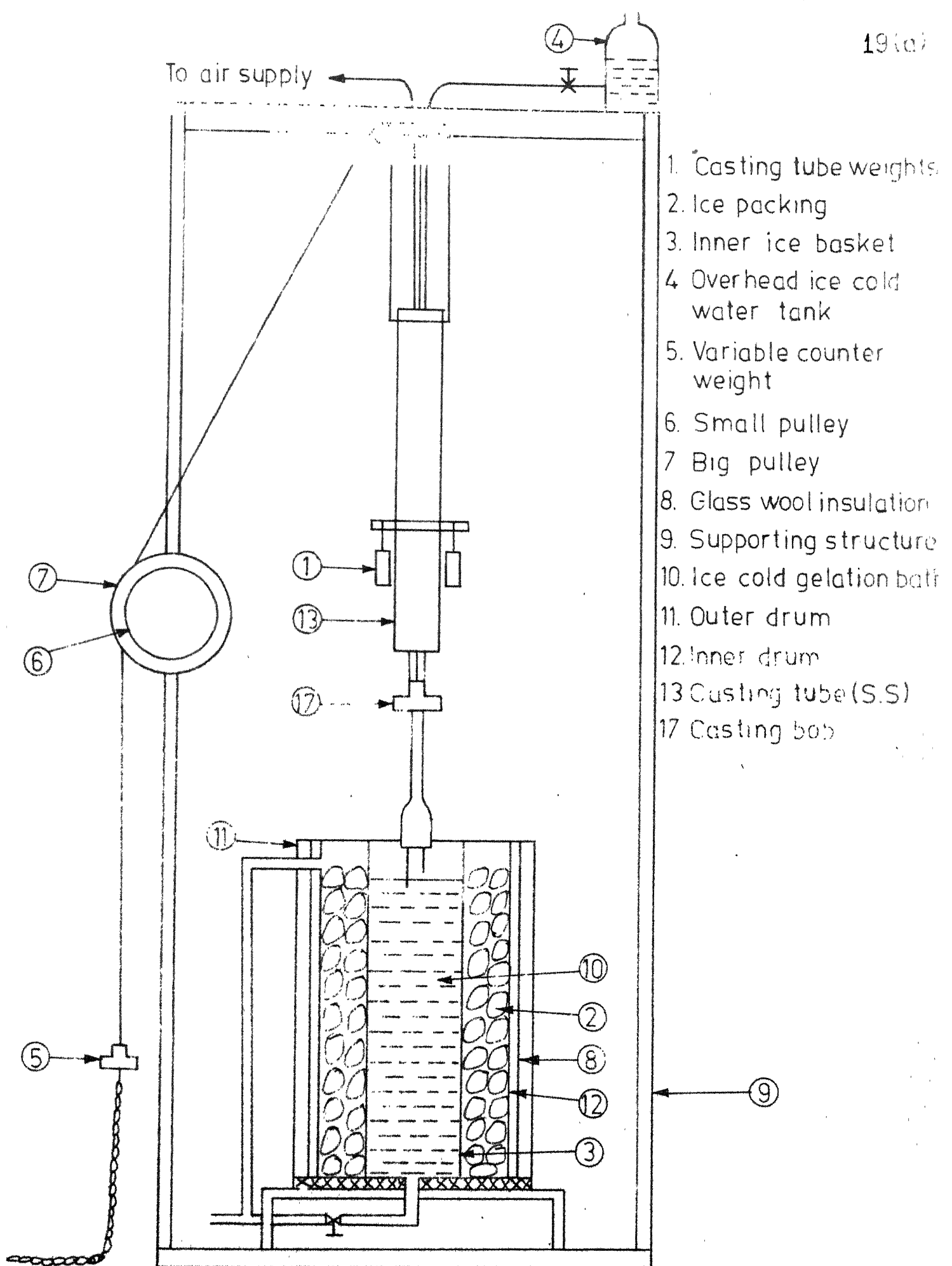


Fig.1(A) - Schematic diagram for casting tubular membranes.

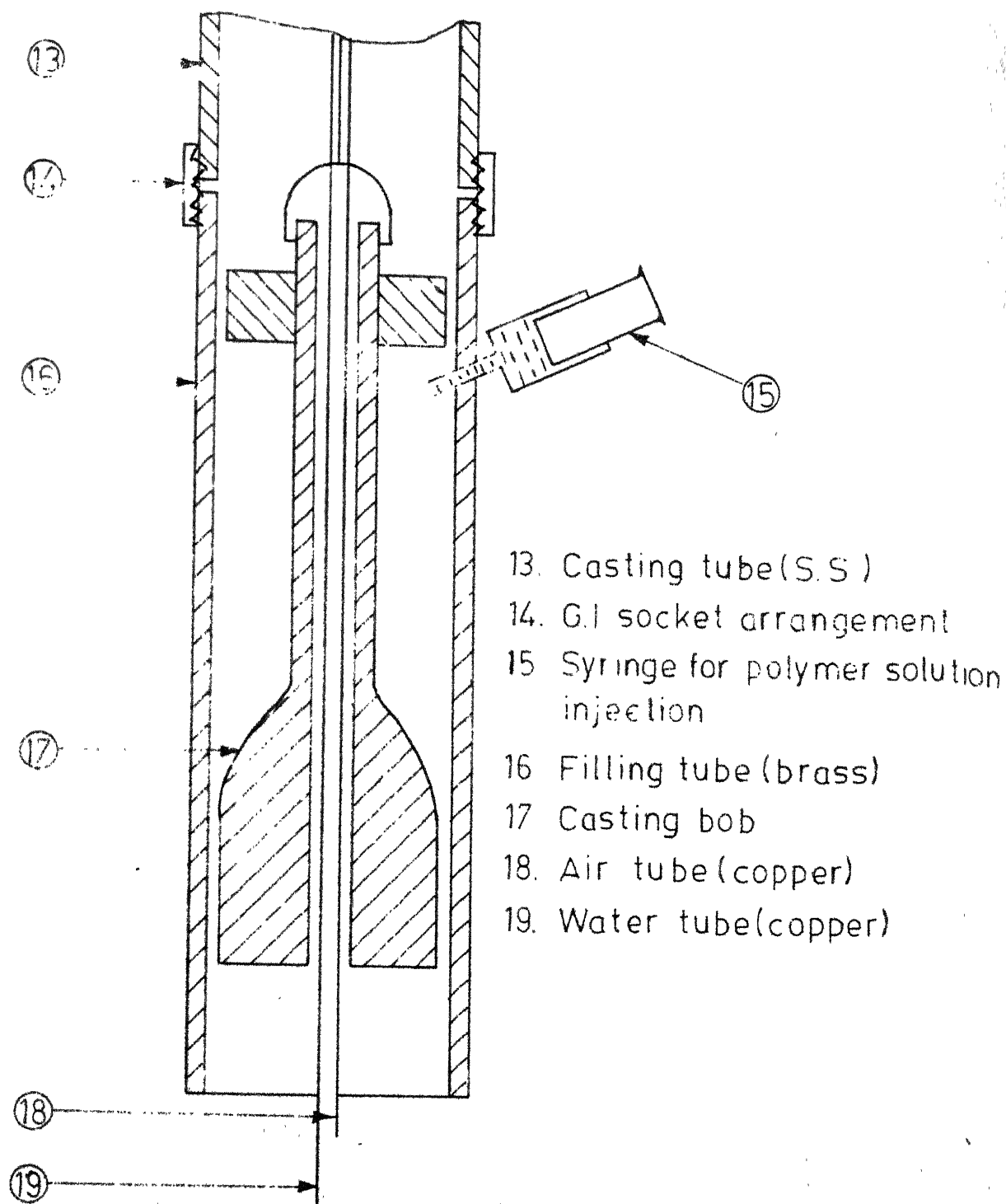


Fig.1(B) - Casting solution filling arrangement.



detached and the casting tube was allowed to fall. Time taken for total fall of the casting tube was noted using a stop-watch. Care was taken to maintain casting speed more than 6 ft/minute. Casting speeds of 6 ft/minute or faster are desirable for void as well as wave mark prevention [18]. And also it was seen that aging of solution is always less than one week to lessen void formation, [18].

(d) Membrane Leaching Tank for Freshly Formed Membrane:

A.G.I. drum of 18 inch dia, 48 inch high is used as leaching tank. Leaching tank sits firmly inside a 23 inch diameter, 48 inch high M.S. drum. The outer drum is used for insulation purpose. Leaching tank is insulated on the outside with a 2 inch thick layer of glass wool. A basket for ice made out of aluminum sheet with 1/4 inch holes in a 1 inch square pitch arrangement is used for holding ice near the wall inside the leaching tank. This basket is 48 inch high and 12 inch in diameter. Ice will be filled in the annulus between the basket and the G.I. drum. An outlet for drainage is provided at the bottom of the leaching tank and an overflow line is provided at the top.

For making a 0-1°C water bath the following procedure was adopted. First the complete annulus between the ice basket and the G.I. drum was filled with ice and tap water is allowed to pass slowly over the ice pieces until about one third of the ice was dissolved, then the tap was closed. Again the

annulus was filled to the top with ice and tap water was passed over the ice. This process was repeated until the tank was filled with water to the required level and temperature. By using this procedure it was possible to bring down the water bath temperature to  $0-1^{\circ}\text{C}$ . If it is desired to maintain very low gelation rate it could be done by mixing sodium chloride in the bath. It will bring down the gelation rate in two ways. Sodium chloride can bring down the bath temperature below  $0^{\circ}\text{C}$  and its presence lessens the driving force for leaching since water concentration outside is reduced [19]. Instead of sodium chloride a little bit of acetone can be added to bring down the gelation rate but it will be very costly.

(e) Air Circulation Arrangement: If the air within the casting atmosphere (below the bob head) gets saturated with acetone during membrane casting, the acetone evaporation from the film and subsequent skin formation will be hindered. To ensure that this did not happen, fresh air was supplied by means of a  $1/8$  inch O.D. copper tube passing through the casting bob and acetone-rich air escaped through the annulus gap between  $1/4$  inch hole in the bob shaft and the  $1/8$  inch copper tubes. The air tube end projected out of the bob. This projected length was adjusted such that the end of it was just above the water surface to ensure that all acetone leaving the film was driven out of gap (acetone vapor is denser than the air). The upper end of the tube was connected to a polyethylene tube which in

turn was connected to an air supplier. For air supplier a bomb type gas storage tank was tried but it was found that it can supply air only for fifteen to thirty minutes. Since it was desirable to cast atleast four to six membranes tubes per day, it was not convenient. It was decided to use a nitrogen cylinder. As very small flow rates were used, flow rates could not be measured but it was checked by dipping the air tube out let in a beaker of water and the rate of bubble escape was about 30-60 bubbles per minute.

(f) Water Circulation Arrangement: Water circulating through the inside of a newly formed membrane under going gelation increases the gelation rate and hence gelation period can be reduced thereby. For this an overhead tank with a flow controlling valve was connected to a 1/8 inch O.D. copper tube by a polyethylene tube and the copper tube was passed through the hole in the bob shaft so as to dip the free end of the tube into gelation bath. The overhead tank was filled with ice-water mixture.

If it is desired to reduce gelation rate some suitable salt like sodium chloride or solvents like acetone can be mixed with the overhead ice water mixture [19]. Our experiments with salt however indicate a quicker color change indicating very quick gelation.

(g) Mechanism for Controlling the Falling Speed of the Casting Tube:

Maintaining a constant casting tube speed helps to promote uniform membrane properties and reproducibility of results. By varying the casting speed, one can alter the air evaporation time for a given gap between the bob and the water and hence the membrane properties. Several drive mechanisms were considered using electric motors, clock work motors etc. It was decided to use a hydraulic drive for simplicity and economy. Further a motor drive with gears driving the casting tube would have produced non-uniform motion. A piston and cylinder from a discarded air-craft shock-absorbent was obtained. The cylinder was provided with a nozzle at the top to provide resistance to the oil flow for obtaining constant speed. Since casting tube falls through about four feet and the piston has to travel only 8 inch, a reducing mechanism of two concentrically mounted aluminium pulleys in the ratio 1:3 was used. Two pulleys of sizes 8 inch and 1 inch were mounted on a brass tube whose ends were supported in two ball bearings. Ball bearings were used for smooth rotation of the shaft which is essential for smooth fall of the casting tube (even small jerky movements can spoil the membrane). In actual operation it was found that this system offers high resistance and fall of the casting tube was very slow. Therefore it was decided to control the speed of the falling casting tube by applying adjustable counter-weights to the shaft discarding

the hydraulic drive mechanism. An aluminum pulley of 4 inch diameter was fixed to the shaft for adding counter weights. The whole system appeared as follows (See Figure 1): casting tube was hung by two nylon ropes (previously it was tried to hang it by three ropes but it had given some trouble in hanging it perfectly vertical) which passes over two smoothly rotating rollers which are fixed on the top of the slotted angle frame. Both nylon rope ends were fixed to the 8 inch pulley. Another nylon rope was fixed to the frame of the 4 inch pulley and wound around its periphery in a direction opposite to that on the 8 inch pulley such that counterweight acts in opposition to the motion of the shaft and required counterweight was attached to the free end of the rope hanging from the 4 inch pulley. A locking arrangement for the shaft motion was made by making small holes on the 8 inch pulley  $1\frac{1}{2}$  inch apart along the circumference and one inch away radially from the edge. To keep the casting tube system in tension, two weights of 600 gms each were attached near the end of the casting tube by means of a strap bracket.

It can be readily seen that casting tube downward motion is opposed by casting solution drag, counterweights, buoyance forces in the gelation bath and bearing resistances. So, the downward acceleration of casting tube,  $a_d$ , can be approximately given by

$$a_d = \frac{(CTW - 2CW)g - \text{solution drag on the tube} - \text{buoyance forces} - \text{bearing resistance}}{(CTW + CW)}$$

where CTW = casting tube weight and CW = counter weight.

The counterweights should be such that after desired velocity is attained by casting tube,  $a_d$  should be zero. i.e.

$$(CTW - 2CW)g - \text{solution drag-buoyance force} - \text{bearing resistance} = 0.$$

$$CWg = \frac{CTWg - \text{solution drag-buoyance force-bearing resistance}}{2}$$

Here, CTWg and bearing resistance are constant, solution drag and buoyance forces are changing with tube fall. So, counterweight must also be variable type to maintain a zero acceleration. This was done roughly by attaching a flexible iron chain to constant counterweight. Chain rest on the ground initially and as the casting tube moves downwards (i.e. counterweight moves up) the chain slowly will be added to the counterweight. In our case, 2 ft long chain, weight 0.3 Kg/ft. was used. It was observed that velocity of casting tube fall was almost constant.

Speed of fall of the casting tube is very much depend on the solution viscosity which is a sensitive function of temperature and composition of the solution. The calibration of average speed with counterweight was not done because

temperature of the solution varies with the time of the day, season etc. and different compositions were used for the casting solutions.

(h) Supporting Structure [17]: The supporting structure is made of slotted angles for the ease of fabrication and flexibility. The structure consists basically of four 10 ft. long pieces of slotted angle which are attached at the top and bottom to 26 inch squares made of slotted angle. This 10 ft. high structure is by itself inherently unstable and so it was stabilised by making the leaching tank rest on the bottom square and attaching the top square to four horizontal struts which are firmly fixed to two beams in the ceiling. The casting bob was suspended from the top of the supporting structure. The various components of the drive mechanism were also attached to the structure.

(i) Membrane Storage Tanks: One end of the brass tube of  $1\frac{1}{2}$  inch I.D. and  $4\frac{1}{2}$  ft. long was closed by brazing a circular brass plate at one end. Only one membrane can be stored in this tube. Membrane was hung from the top of the above tube using a clip into 0.4 per cent formaldehyde solution [18] prepared by using distilled water and 40 per cent formaldehyde solution, to save the membrane from bacterial attack.

When three to six membranes were being cast in a single day, a big membrane storage tank was required. So, a plexiglass tube of 5 in. diameter,  $4\frac{1}{2}$  ft. long was used.

obtained and one end was closed by a circular plexi-glass plate. At the top an arrangement was made to hang the membrane tubes by clips and the top was covered with a lid to prevent evaporation of storage water. This can accommodate more than ten membranes.

#### B. TUBULAR MEMBRANE TEST CELL AND MEMBRANE MOUNTING:

In this section, the test cells used for tubular membranes are described first. Then the membrane mounting procedure is given, with the concluding section dealing with turbulence promoters and annular arrangements.

(a) Tubular Test Cell: The tubular membrane test cell consists of a metallic supporting tube with nylon backing cloth, end seals with connections and permeate collection arrangements. A supporting tube is needed because the membrane to be tested must be supported on the back side as unsupported membranes cannot withstand more than a few psig of pressure without damage. These support tubes have to be porous to allow permeate to come out without much pressure drop. Several materials are available for support tubes e.g. porous stainless steel tubes having 1 $\mu$  pores (made by powder metallurgy technique), resin bonded sand tubes, fibre glass reinforced epoxy tubes, solid metal tube with a few holes in the wall and having a few wraps of porous cloth on the inside. Except the last one, none of the other materials have been tried out for the present experiments because of the constraints of cost and availability.



Porous reinforced epoxy tubes could not be procured for the present work but are expected to arrive soon. The advantages of a porous fibre glass reinforced epoxy tube is that in-situ casting is possible i.e. the same tube that is used for casting acts as a pressure vessel also supporting the membrane.

Proper end sealing arrangements at the end of the tubular membrane are absolutely essential to prevent leakage of the feed brine to the back sides of the membrane. Otherwise mixing of concentrate with permeate nullifys any separation achieved. Several types of end seal arrangements have been used by previous workers [6], [13]. One of the interesting configurations [20] is the quasi - 'self-sealing' owing to the difference in the moduli of elasticity. A reinforced plastic seal tube intrudes into the RO membrane tube and this tube itself will expand under pressure thus increasing the compression on the fit which effects the seal. As this type of seal could not be procured, some other types of seals were used.

As the membrane performance varies significantly along the length of the membrane even under controlled conditions of casting [6] due primarily to concentration polarization, it is of interest to find out membrane performances at different points and thereby calculate the brine concentration on the membrane at different points. This is possible by making compartments on the outside of the supporting tube and collecting permeate in each compartment separately. Care was

taken to keep the cell perfectly horizontal during testing so that no permeate from one portion of the membrane comes into the other portion of the collector tube. In all the test cells to be described such an arrangement was made and used.

The description of our test cells are as follows:

(a) Test Cell-1: Adhikari and Gupta [17] designed this test cell but later this was not found to be very suitable. Here a copper tube was used as the supporting tube. It was 24 inch in length and had  $7/8$  inch I.D. and 1 inch O.D. with holes of  $1/16$  inch diameter all along the length at regular intervals of 3 inch longitudinally and four equidistant holes on the circumference at each longitudinal position. It contains end fittings as shown in Figure 3. The end seal consists of a rubber grommet inserted over a brass expansion tube which has to be pushed inside the membrane tube at both the ends. Rubber grommet was given a taper such that the initial portion goes very easily into the membrane and the last  $3/16$  inch of the membrane is pressed hard. The expansion tube is fixed firmly to the end flanges by bolts and nuts. The end flanges are made of ordinary brass. Problems were faced in choosing a proper material for rubber grommet. In the first attempt, ordinary rubber was machined (during machining, rubber had to be kept at very low temperature using liquid  $N_2$ ) with difficulty but unfortunately it was found to be unsuitable. A silicone rubber tube inserted

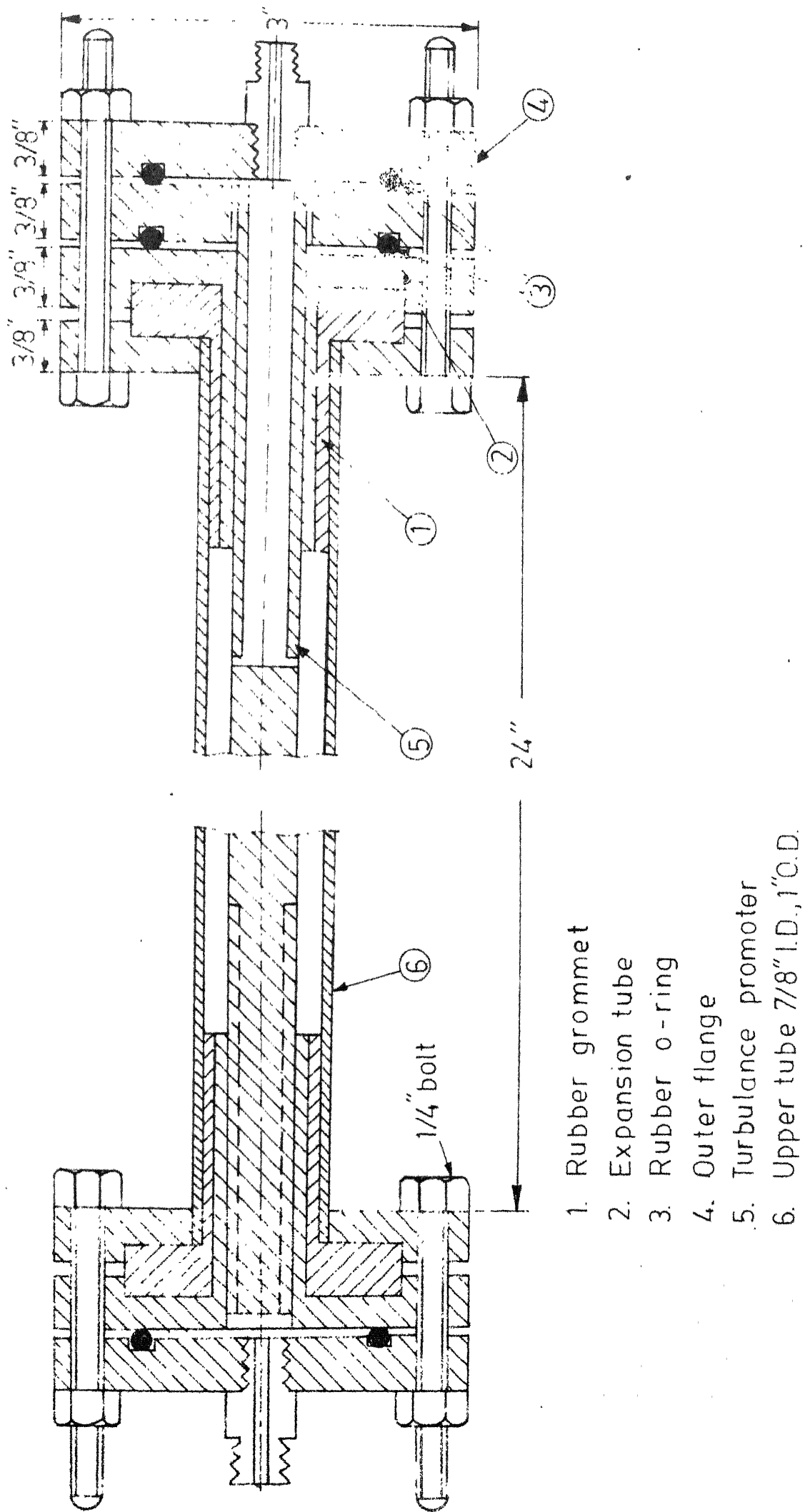


Fig. 3 - Sectional view of tubular membrane test cell 2

over expansion tube was ground on the lathe, using emery paper. to the required dimensions. This did serve the objective. Once seal had been made, expansion tube cannot be taken out without damaging the membrane end, for inserting turbulence promoter rods etc. So, one of the end expansion tube had to be redesigned to insert the turbulence promoter rod without taking out the expansion tube. In this design, the thickness the rubber grommet (gap between the expansion tube and the membrane) had to be only 0.06 inch. To machine a rubber to such a small thickness for the rubber grommet, lot of problems were faced in material selection as well as in machining. A variety of materials were tried including rubber balloons but none succeeded. Wrapping the tube with teflon to required thickness was found to serve the purpose. Finally this test cell was rejected totally due to the following difficulties

(a) The first problem was insertion of rubber grommet in to the membrane tube to effect the seal. While pushing the rubber grommet into the membrane, the membrane itself was getting pushed out by rubber grommet. To eliminate this, it is first necessary to plasticize the end of the membrane with a 9:1 by volume mixture of n-propyl-alcohol: triacetin [6] after which the rubber grommet is pushed in. Even after applying the plasticizing agent, sometimes the seal failed due to pushing of the membrane. The bad effect of plasticizing is that it spoils the membrane skin structure at the ends and hence

membranes perform poorly at the ends. With experience, the extent of plasticizing required can be minimized but cannot be eliminated totally.

(b) The other difficulty in this test cell was too much gap (0.035 inch) between the membrane tube and the supporting tube. This meant twelve-wrappings of 0.003 inch thick nylon cloth has to be given to the membrane so that the nylon wrap O.D. matches with the test cell metallic tube I.D. Due to the large number of nylon wrappings the membrane was developing folds and tearing off at the folds during the high pressure testing thereby giving very poor results at those particular points.

These difficulties may be overcome by modifying the design suitably. Reducing the rubber grommet diameter such that it goes freely inside the membrane without the necessity of plasticizing agent and using a different material which has low elasticity of modulus for expansion tube which will expand under pressure to the required extent to effect the seal. A very good end sealing arrangement has however been used by Sourirajan et al. [10]. This has several advantages over the above mentioned design. This design was adopted with some modifications for the fabrication of test cell 2 which will be described shortly.

For tapping permeate at different portions of the support tube,  $1\frac{1}{2}$  inch diameter circular plexi glass plates with 1 inch central hole were inserted over the support tube and fixed at the required position by using araldite. A  $1\frac{1}{2}$  inch I.D. plexiglass tube was pushed over the plexiglass partition plates and made leak proof from one compartment to other by joining partition plate edges with the plexiglass tube wall using trichloroethylene. A number of 0.2 inch diameter holes in a line and one per each compartment were made on the plexiglass tube. About 2 inch long polyethylene tubes were inserted into these holes and fixed thereby using araldite. Permeate was collected in 100 ml Corning conical flasks. In this test cell, six equal partitions were made and each covers 9.23 square inch of membrane area which corresponds to 3.67 inch long membrane of 0.8 inch internal diameter. Here 100 ml. of permeate collected per hour per each compartment equals to 10.0 GFD (conversion factor calculation is shown in the Appendix ).

(b) Test Cell 2: In this test cell, a stainless steel tube of 0.828 inch I.D., 1.0 inch O.D., 24.1 inch in length was used as a supporting tube. The stainless steel tube was used only on the basis of I.D. requirement (only this tube is having the required I.D. to match the casting tube I.D. plus the membrane thickness plus two wraps of nylon cloth).

Holes of  $1/32$  inch diameter were drilled at intervals of 1 inch longitudinally and two holes were made along any diameter at each longitudinal position. The line joining these two holes changes by  $90^\circ$  with adjacent holes. This tube has a total of 32 holes. Both ends of the tube were threaded on the outside for fitting a sealing assembly.

The test cell assembly uses O-ring sealing technique and simple end fittings, requiring no flarings or flanges to the support tube and no softening, plasticizing, flaring, bending or folding for the membrane ends [10]. As shown in figure, the end fitting consists of a seal tube (6) with an O-ring shoulder at one end and an adjusting nut (5), a Swagelok joint fittings (10) on the other end, turbulence promoter seat, two sleeves (8) (one smaller and the other bigger) and a retainer inbetween with O-rings placed on each side of the bigger sleeve as shown in the Figure 4. The diameter of the shoulder is slightly less than that of tubular membrane. The two O-rings (7 and 9) are initially in the relaxed position in the tapered necks of the seal tube. The seal tube with O-rings in relaxed position and sleeves and the retainer, adjusting nut placed in position is kept as a single assembly ready for use.

For permeate collection, an arrangement was made similar to that of test cell 1 except that only seven partitions were made. Partition No.1 and No.7 cover equal lengths at each end and each corresponds to 3.5 inch long membrane i.e.

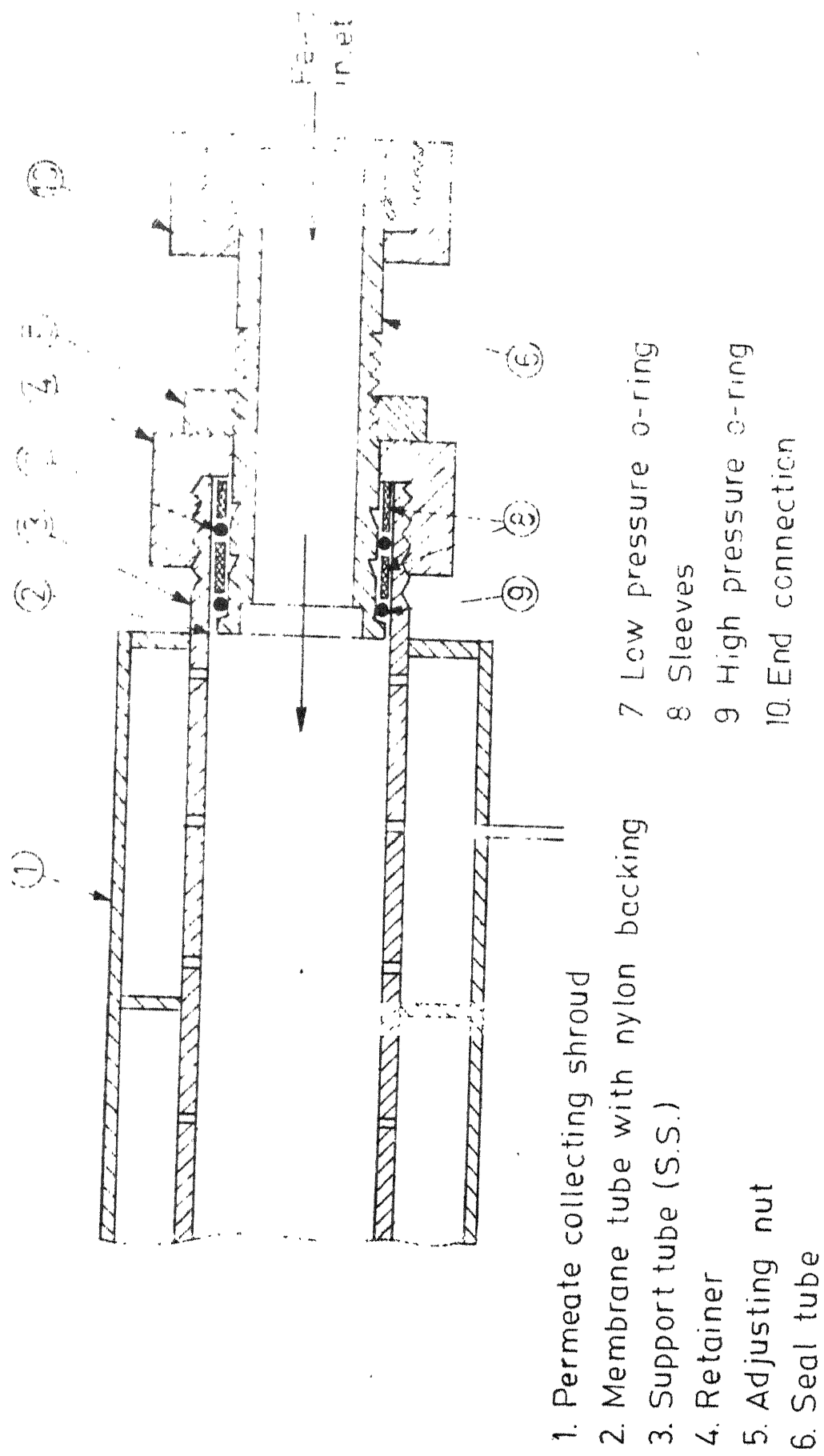


Fig. 4 - End sealing assembly of test cell 2



8.8 sq. in. in membrane surface area. Remaining partitions are of equal size and each covers 7.54 sq. in. area corresponding to 3 inch long membrane. In the case of 1 and 7 partitions 100 ml per hour of permeate collected is equivalent to 10.46 GFD and for the remaining portions 100 ml per hour permeate is equivalent to 12.1 GFD.

(b) How to Assemble a Membrane for Testing? Assembling the membrane and test cell for high pressure testing consists of wrapping the membrane with fine nylon cloth (or similar cloth), cutting the membrane to the length of the support tube, pushing the membrane with nylon cloth into the test cell and sealing the ends. Nylon cloth backing is required to save the membrane from possible ruptures at the location of the supporting tube holes. Nylon cloth used is 180 mesh size and of thickness 0.003 inch. Supplied by the General Distributors (International), Calcutta. The nylon cloth extends along the entire length of the membrane which is thus equally supported throughout its length including the area at the end where the seal is accomplished unlike the previous arrangement in Test Cell 1. As the membrane tube is flimsy, a support tube is required during the wrapping of nylon cloth over it and pushing of the membrane into the test cell. This supporting tube is a glass tube of 0.75 inch O.D. and 4 ft. in length. Care should be taken to keep the membrane always wet during the entire period of operation. This was done by using

polyethylene water bottles.

In the case of test cell 1, the procedure for assembly was as follows: Inserting the membrane tube over glass supporting tube and wrapping with nylon cloth (12 wraps are required). Then the nylon cloth membrane is tied at one end by a fine nylon thread. This end was first pushed into the test cell. The membrane length in the test cell was adjusted such that membrane end coincides with the supporting tube end. Then the membrane is cut to the length of the support tube length exactly. After applying the plasticizing agent (about 1 cc) on the membrane end, the rubber grommet which is already on the expansion tube is pushed in very carefully. Expansion tube with other end connections was fixed tightly to the end flange of supporting tube by using three M.S. bolts and nut (which must be tightend equally). After sealing the other end also, the test cell was ready for heat treatment of the membrane.

In the case of test cell 2, the membrane was loaded into the test cell similarly as above. Here only two nylon wraps are required and so, the dimensions of the cloth will be 24.1 inch in length, 5.1 inch wide. After the membrane is cut to the test cell size, the seal tube assembly is inserted freely into the tubular membrane in the cell. The retainer is tightened first and then the sleeves are pushed toward the membrane to bring the O-rings from this tapered

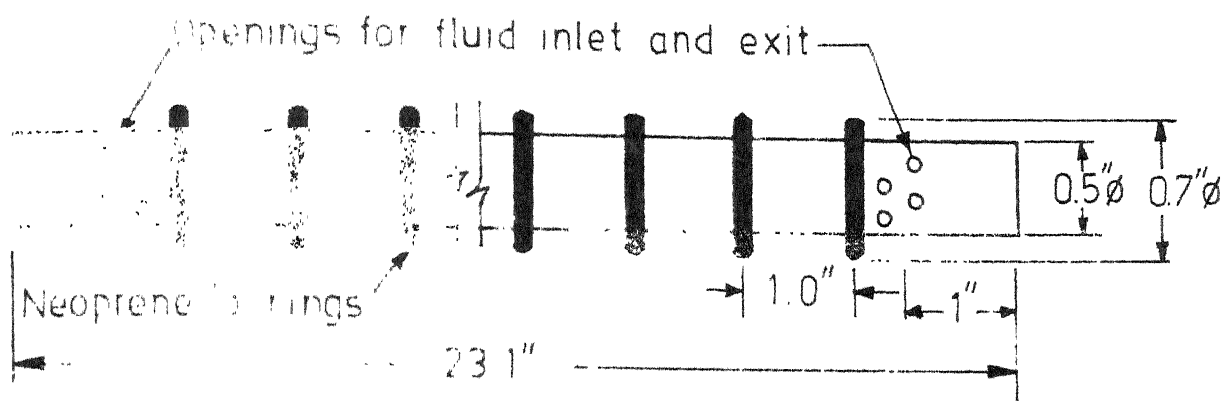
necks to the required sealing positions. Finally sealing was accomplished by tightening the adjusting nut to the required extent. The test cell could now be used for heat treatment of the membrane.

### (C) TURBULENCE PROMOTERS:

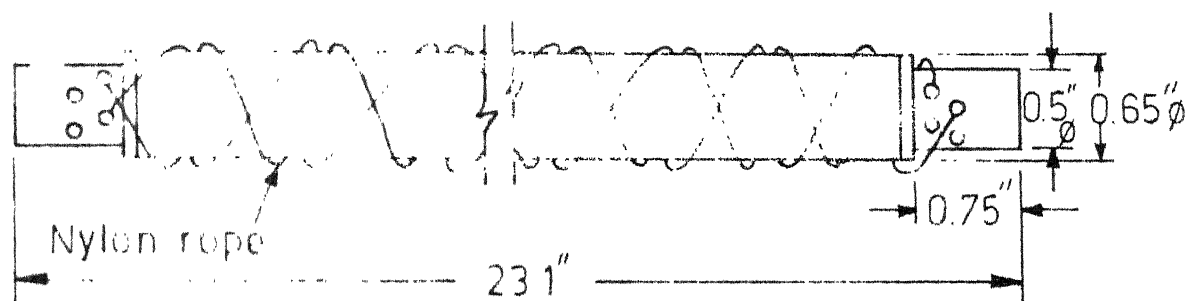
One of the severe problems encountered in RO membrane separations is concentration polarization. During the separation process, a concentration boundary layer develops on the membrane surface. At very low Reynolds numbers salt concentration at the membrane surface may become 10-20 times more than the bulk concentration which decreases drastically both the water flux and the percentage salt rejection. This suggests that a high mass transfer rates of the salt from the surface to the bulk stream must be achieved. A common practice is to place hydrodynamic obstructions or turbulence promoters in the flowing stream near the wall to break up the polarization layer which accretes at the membrane surface. Very little work on the turbulence promoters are available in RO literature and for better working conditions in RO modules a considerable amount of work is necessary. The objective of our work with regards to turbulence promoters was to see how well some types of turbulence promoters effected an improvement in overall performance, without going into (a) an economic balance which results from the increasing requirement for pumping energy or (b) the development of improved turbulence promoters.

Two different types of turbulence promoters were used in this work and are shown in Figure 5. The first one (code named TPI) is a 0.5 inch O.D. copper tube whose inside is closed by tightly pushing a brass piece and having O-rings placed on its O.D. at a equal spacing of 1 inch. The dimensions of the O-rings used are 0.625 inch O.D. and 0.425 inch I.D. The second turbulence promoter (code named TP2) is a 0.65 inch O.D., copper tube with 0.033 inch nylon wire double wound helically in opposite directions. In an attempt to see what would be the effect of an annulus only, a 0.75 inch O.D. copper tube was used in the arrangement code named TP3. After a membrane tube has been mounted in test cell 2, introduction of any annulus with or without turbulence promoters requires the opening of one of the end seal assemblies and introduction of the annulus whose ends must fit exactly in the seal tube assembly as shown in Figure 4.

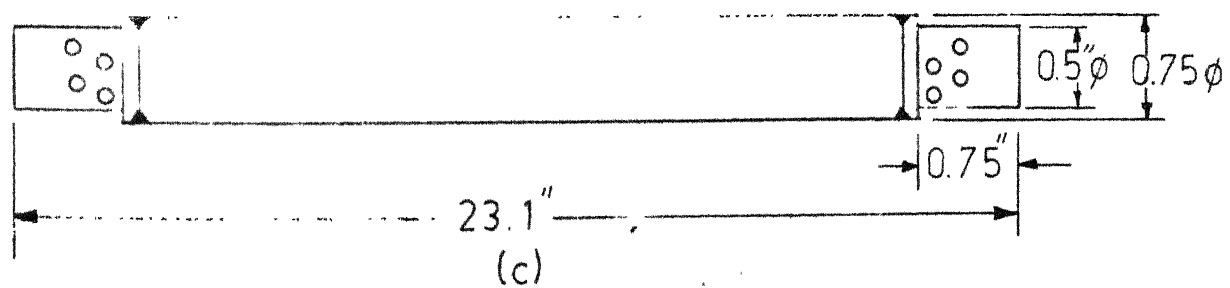
C. Heat Treatment: Unheat treated (as cast) membranes are very porous and yield very poor salt rejections. So, membranes are heat treated to get desired membrane skin structure. The temperature of heat treatment sets the level of salt rejection produced by the membrane. A higher range of temperature of heat treatment produces high salt rejection but a membrane with low water flux and vice-versa. According to Wasikwski [25] in the unheattreated membranes, almost all cellulose-acetate molecules are intramolecularly hydrogen



(a)



(b)



(c)

Fig. 5 - Various types of annulus rods with turbulence promoters.

bonded and during heat treatment above  $65^{\circ}\text{C}$ , the formation of intermolecular bond between two neighbouring CA molecules in the membrane during heating, brings the segments of these two molecules closer together resulting in a decrease in the pore size, in the surface layer of the membrane. To obtain a desired high level of salt rejection, some optimum minimum annealing period is necessary beyond which no appreciable change in the membrane performance will be observed. Membranes in this study were heat treated in the range of  $80-86^{\circ}\text{C}$  for about 10 minutes.

The assembly is heated by internal flow of hot water to the temperature required. During heating a pressure of 7 to 8 psig and during cooling a pressure of about 9-12 psig are maintained. Slight pressurization during heating operation is critical for the success of the membrane tube assembly. This requirement stems from the fact that heating the membrane causes an unrestrained membrane to shrink by a few per cent (contrary to the thermal expansion one ordinarily expects upon heating) and from the experimentally determined rule that heat treated membranes should not thereafter be subjected to tensile stresses because this causes undesirable modifications of the appropriate microstructure in the membrane skin set-up during heating. If tubular membranes were heated without restraint they would contract appreciably away from contact with its concentric supporting structure. Under high pressures

of subsequent desalination service the tube would then expand causing disruption of its microstructure or even its microstructure. To avoid this, the heating phase is conducted at a pressure slightly higher than the heated yield strength of the material. Therefore, the membrane tube will flow plastically during the initial parts of the heating phase to the full diameter of its supporting structure, after which further heating causes it to attain the microstructure desired for the desalination requirement. Finally the subsequent cooling phase is preferably conducted at a pressure slightly above the ambient temperature yield pressure, so that no true thermal contraction occurs during cooling. By these means, the completed membrane tube is subjected to compressive stresses only, during the subsequent pressurization occurring during desalination service.

The heattreatment loop is as shown in the Figure 6. It consists an over head tank for keeping cold water, a thermostat (Ultra-Thermostat Type NBE of VEB PRUFGERATE WERK MEDINGEN S /T<sup>2</sup> FREITAL, German Democratic Republic) and an Alfa Laval S. Steel centrifugal pump of 0.75 h.P. The sequence of operations are as follows: cold water was circulated through the by-pass keeping the valves B, C and E open and valves A, D, F closed. The valve D is slowly opened and then valve C is slowly closed, to switch off cold water circulation from by-pass line to the membrane tube side. This by-pass arrangement

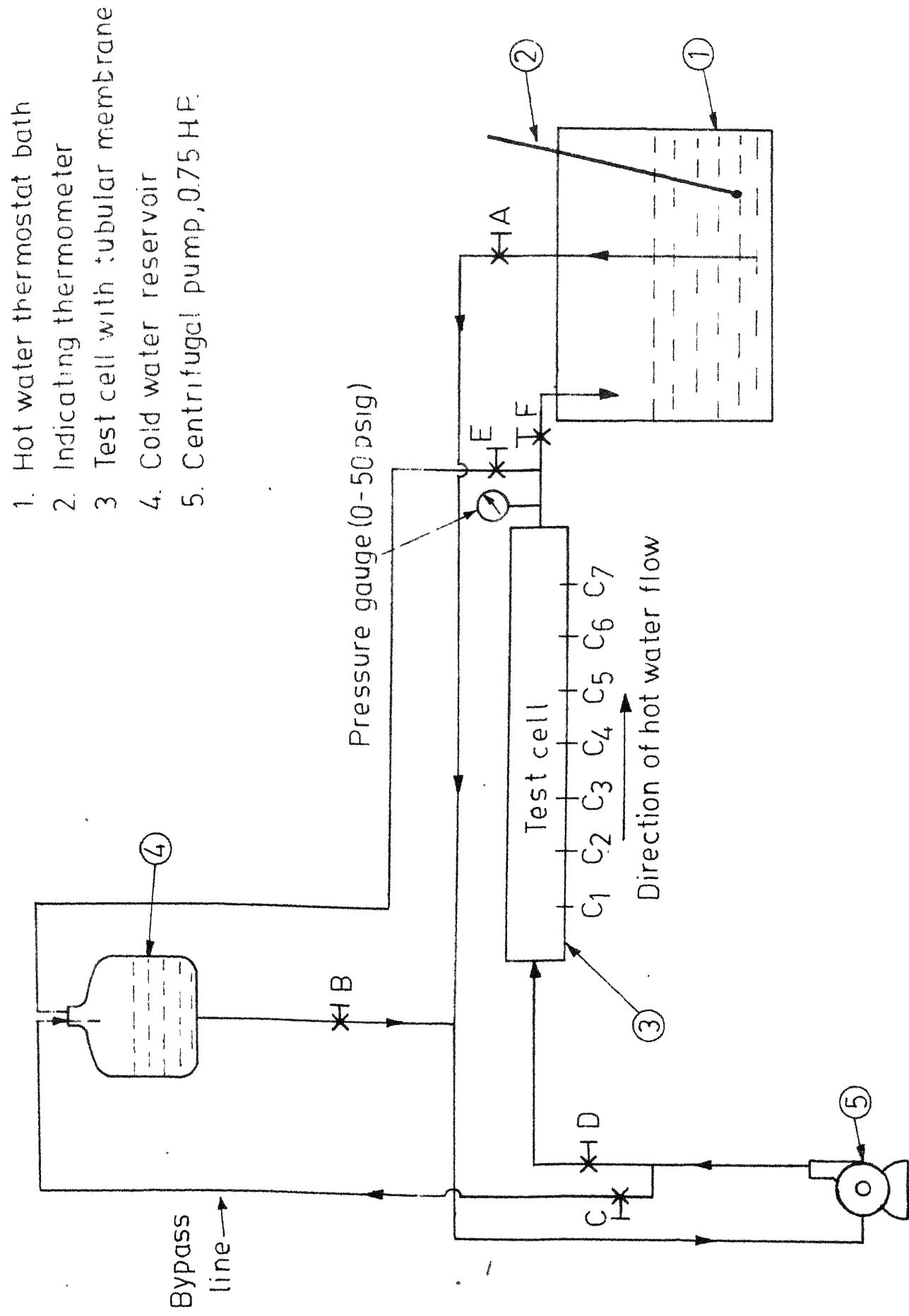


Fig. 6 - Schematic for tubular membrane heat treatment flow loop.



is used to prevent the membrane from experiencing sudden stresses due to sudden pumping, which may damage the membrane structure. By adjusting the outlet valve E the inside pressure was maintained at 7-8 psig. Next valve B is closed while simultaneously valve A is opened. Now hot water from the thermostat tank passes through the system. The hot water outlet is gradually charged by slowly closing the valve E and simultaneously opening the valve F to the same extent. Care should be taken to see that the inside pressure does not fall below 8 psig, at any time during this process of swifting outlet. This heating is continued for 5-15 minutes at the required temperature. If the membrane is restrained at the ends during heating there is a possibility of membrane damage at the membrane ends. This can be prevented by loosening the end seals which can be done by loosening the adjusting nut in the test cell before hot water comes in. After first one minute of heating, seals are tightened again by adjusting nut to prevent hot water wastage. After required period of heating, the inside pressure is increased to 9-12 psig and inlet and outlet valves slowly changed from A to B and F to E respectively. while keeping the pressure in 9-12 psig range, Now cold water passes through the membrane which should be continued for atleast 5 minutes. During cooling process also end seals can be loosened to save the membrane from any strains at the seals. Ordinary tap water was used for both cold and hot waters.

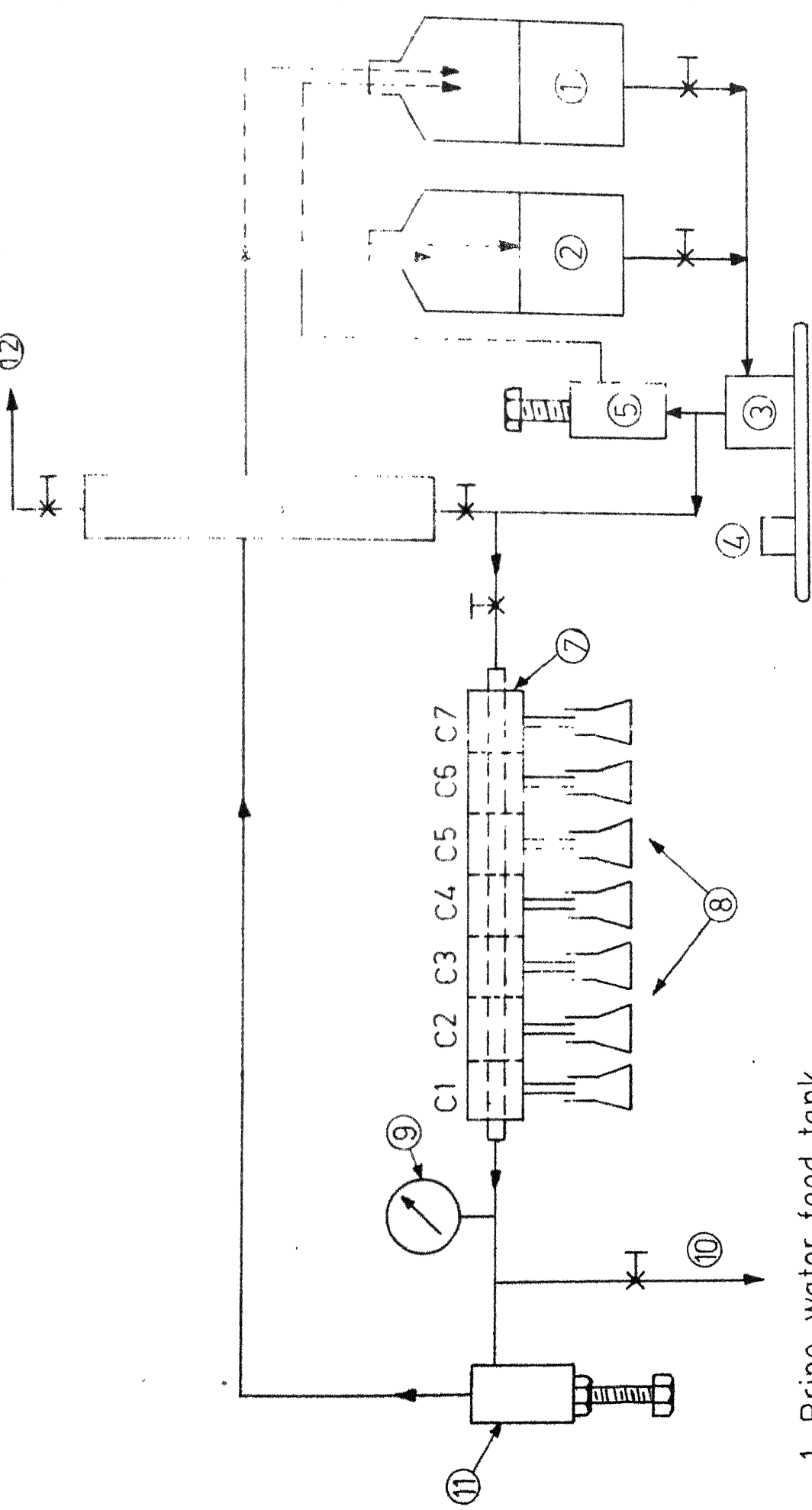
In the above heattreatment the membrane is restrained. from contraction during heating but unrestrained heating may give more compact skin structure. So, a plexiglass heating trough was fabricated. It is a plexiglass tube closed completely at one end, other end can be tightly closed after placing membrane inside and provided with inlet and outlets which can be connected to pump outlet and thermostat inlet respectively. A 28 inch<sup>long</sup>/membrane is cut and inserted over a 17 mm dia glass tube (it should not be restrained by glass tube during heating or cooling) and placed in the above heating trough and hot water is circulated through it for 10 to 15 minutes and then cold water is circulated for 5 minutes. As it is allowed shrink freely during heat treatment it changes its O.D. and I.D. considerably. So, a small portion of membrane tube is cut and its O.D. and I.D. measurements are made by travelling microscope. Now, number of nylon wrapping required is calculated. Not a single membrane was heattreated in this manner because the end sealing assembly which is designed for 0.8 inch I.D. membrane cannot be used for this membrane. However, the membrane heattreated like this was tested on flat membrane cells, which indicated us that unrestrained heat-treated membranes gives better performance.

#### (D) HIGH PRESSURE TESTING LOOP :

The flow loop shown in Figure 7 consists of feed tanks, high pressure pump, pressure safety valves, surge tank, tubular

reverse-osmosis cell with tubular membrane, pressure gauge, purge line and a back pressure regulator. A description of this loop is given in reference [14].

Membrane Testing: After the heat treatment of the membrane, the test cell is connected in the high pressure loop as shown in Figure 7. The sequence of operations is described in the reference [14]. Unless otherwise stated the membranes are pressure treated for one hour at 300 psig with distilled water and the permeate was collected for the last fifteen minutes of the operation. This gives the pure water permeability (PWP) at 300 psig. After releasing the pressure, the feed was changed to brackish water containing 5000 PPM salt. The permeate flux and salt content was measured by collecting the permeate for a fixed time and analyzing it for salt. The performance measured and reported were during the period from 15 to 30 minutes of operation. The salt content was measured by titrating against standard silver nitrate solution using potassium chromate solution as indicator. Salt concentration can also be calculated by measuring solution conductivity with the help of Direct Reading Conductivity Meter Type 303 of Systronics Inc. The standard plots of conductivity versus concentration at different temperatures were available in reference [22] and are given in Figure 10 in Appendix. The difference between the two salt analysis methods was usually less than 2 per cent of salt content, corresponding



1. Brine water feed tank

2. Distilled water tank

3. High pressure reciprocating pump

4. Motor

5. Safety valve

6. Surge tank

7. Tubular membrane test cell

8. Collection flasks

9. Pressure gauge

10. Purg tank

11. Back pressure regulator

Fig. 7 - High pressure membrane test cell

to an error less than 0.2 per cent in percentage salt rejection. If the temperature of the feed is different from 25°C, FWP and flux were corrected to 25°C using the inverse proportionality relation between flux and water viscosity at any temperature as shown by Sourirajan [21] and generally followed in the reverse-osmosis literature.

Our scheme of experiments with each membrane were as follows (a) without turbulence promoters: Three experiments at three different flow rates, roughly at 500, 600 and 750 cc/minute. (b) with turbulent promoters I: two experiments at two different flow rates roughly at 500 and 750 cc/minute. (c) with turbulence promoters II: two experiments at flow rates 500 to 750 cc/minute. (d) with turbulence promoter III: two experiments at flow rates 500 and 750 cc/minute.

All the above experiments were conducted at 250 psig and 5000 ppm feed solution. The flow rates could not always be maintained the same in all membranes due to difficulties in exact stroke length adjustments .

Flow membranes were also tested for 7500 ppm at a operating pressure of 350 psig at a pressure treatment of 400 psig and for 10000 ppm at an operating pressure of 400 psig, at a pressure treatment of 450 psig.

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## CHAPTER 3

### RESULTS AND DISCUSSION

In this experimental investigation, tubular CA membranes were cast from the best casting compositions of Pandurangaiah[14] and Ghosh [23] allowing only low air evaporation periods which varied between 0.5 seconds and 2.5 seconds. The performance of these tubular membranes have been obtained by conducting desalination runs at 250 psig with 5000 ppm sodium chloride solution simulating typical brackish waters. Unless otherwise stated, all membranes were pressure treated with distilled water at 300 psig for 1 hour prior to desalination runs to stabilize the performance. Almost always the new test cell 2 was used for all the data reported. The results are presented and discussed in the following order:

- (A) Physical nature of cast and gelled CA tubular membranes;
- (B) Averaged performance of tubular CA membranes with various annular arrangements;
- (C) Local membrane performances along the membrane tube length;
- (D) Effect of air blowing on membrane performance;
- (E) Comparison of tubular membrane performance with that of flat membranes cut out from the same membrane tube

(F) Concentration polarization as a function of  
membrane tube Reynolds Number.

It needs to be emphasized here that membrane performances are to be judged only in terms of the water flux and the per cent salt rejection the calculation methods for which are available in Appendix .

A. Physical Nature of Cast and Gelled Membranes:

After a certain amount of trial and error, tubular CA membranes free from any visual defects were produced for this study. Initially some membranes had some ring marks which were primarily due to the jerky motion of the casting tube or due to accidental disturbances to the falling casting tube if its speed of fall was low. The ring marks are highly undesirable since they are potential regions for salt leaks and are indicative of total rupture of the membrane skin. These are also called 'wavemarks' in literature and their nature and the manner of formation has been discussed in detail in references [18] and [22]. In general, CA membranes (of thickness 0.008 - .012 inches) cast at a speed of more than 2 feet/minute from casting solutions having a viscosity greater than 30 poise are found to be free of wave marks [18]. Table 1 contains the thickness and outer diameter of various sections of membranes cast in this work. Small variation in thickness of the membrane was present both along the circumference of the membrane tube as well as along the tube length. Such small



TABLE 1

## VARIATION IN TUBULAR MEMBRANE THICKNESS

Thickness Measurements<sup>+</sup> of  
Tubular Membrane<sup>++</sup>

AND DIAMETER

+++ Membrane O.D. Measurements of  
two different compositions

S. No.	Distance from one end in longitudinal direction inches	Thickness variation along circumference			Location along various radial positions	a O.D. inch	b O.D. inch
		t <sub>00</sub> inch	t <sub>120°</sub> inch	t <sub>240°</sub> inch			
1	0	0.008	0.008	0.008	1	0.816	0.830
2	15	0.009	0.008	0.008	2	0.808	0.814
3	30	0.010	0.007	0.006	3	0.825	0.812
4	46	0.015	0.005	0.004	4	0.843	0.818
					averaged diameter	0.822	0.818

+ These measurements were taken by a Micrometer without pressing the membrane.

+++ Measurements were taken by a Travelling Microscope

Composition of casting solution: 30 per cent Ca, 25 per cent F, 45 per cent AC or the membrane

a Casting solution composition: 30 per cent Ca, 20 per cent F, 50 per cent AC, 2.44 gm Mg(ClO<sub>4</sub>)<sub>2</sub> · 6H<sub>2</sub>O

b Casting solution composition: 30 per cent Ca, 25 per cent F, 27 per cent AC, 18 per cent D.

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variations are not expected to affect the membrane performance as long as the membrane skin is unaffected. In the initial 3 - 2 inches of the membrane formed, one side of the membrane was consistently very thin compared to the required thickness. The extent of thinning was finally reduced by centring the casting bob exactly in the casting tube before casting proceeded. Any membrane length with too much thinning was rejected for testing purposes. The variation in membrane diameters reported in Table 1 is too much and is a result of the difficulty in estimating true diameters from a travelling microscope.

B. Average Tubular Membrane Performances at 250 psig with 0.5 weight per cent Brine Feed:

Since the maximum flow rate of the positive displacement pump UMPN-35 used in these studies was only 800 cc/minute corresponding to a Reynolds number of 9.28 with respect to the membrane tube internal diameter, it was necessary to incorporate some annular arrangements inside the membrane tube. Such arrangements have been already described in Chapter 2 Section C and tests were carried out at two flow rates only 500 cc/minute and 800 cc/wt.per cent/min. The various flow configurations are: (a) No annulus (None); (b) Annulus 1 (TP1) - This was a 0.5 inch O.D. copper tube with 0.1 inch high circular turbulence promoters placed at a spacing of 1 inch apart; (c) Annulus 2 (TP2) - This was a 0.65 inch O.D. copper

tube having 0.033 inch high turbulence promoters wound in double helical pattern; (d) Annulus 3 (TP3) - This was a bare 0.75 inch O.D. copper tube with no turbulence promoters.

The averaged performances of tubular CA membranes cast from various compositions and different casting conditions with various annular arrangements are given in Table 2. The new test cell 2 was used for all of these membranes. The results clearly show that all annular arrangements which reduce flow cross sections and increase wall shear rate are producing much better membrane performances than that is possible without any annulus (None). This result merely confirms the existence of severe concentration polarization in the tubular membrane without any annulus. Among the various arrangements of annuli with turbulence promoters, arrangement TP1 with 'O' rings on annulus rod performs better than TP2 with helically wound wires. This is to be expected since electrochemical mass transfer studies in channels with ring turbulence promoters (perpendicular to mean flow) has been found by Leitz [24] to be the most efficient in improving mass transfer coefficient. Since the O.D. of the annulus of TP1 is less than that of the O.D. of TP3, the performances of membranes with these two arrangements should not be compared. In TP3 the gap is very small so that the membrane performance is almost comparable to that with TP1. Since the O.D. of TP2 annulus is much larger than that of TP1 and yet the performances

TABLE 2

PERFORMANCE AND CASTING DATA FOR CA TUBULAR MEMBRANES WITH OR WITHOUT

AIRLINES CUM TUBULE PROTECTOR

S. No.	Membrane Code No.	Composi- tion of casting solution	Conditions of casting and testing	AVERAGE PMP GFD	TP0 % R	TP1 % R	TP2 % R	TP3 % R
1	2	3	4	5	6	7	8	9
1	M-2 14-5-76	32 22 27.6 18.4	2.15 7.3 105 80 35 500	28.2	548 15.2 33.3	357 18.7 58.5	321 18.8 46.3	- - -
2	do	do	2.15 7.3 105 80 35 600	do	695 13.8 30.7	428 19.0 64.4	385 18.2 52.0	360 18.9 54.8
3	M-4 14.5.76	32 22 27.6 18.4 2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	0.95 10.4 115 77 35 300	25.0	584 14.6 22.0	357 16.8 54.8	321 17.2 48.5	300 17.7 56

Table 2 (contd)

1.	2	3	4	5	6	7	8	9
4	M-7 4.6.76	32 22 27.6 18.6 2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	0.95 9.5 60 81 35 570	22.2	664 11.1 42.7	407 14.9 68	367 15.0 63.0	360 16.5 63.7
5	M-8 4.6.76	32 22 27.6 18.6 2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	0.95 9.2 6.0 85 34 500	34.7	584 16.6 21.2	357 12.0 51.0	321 12.8 52.1	300 15.6 48.4
6	do	do	0.95 9.20 60.0 85.0 34.0 750	do	670 11.8 33.8	535 13.4 55.3	481 16.6 40.8	450 16.6 48.0
7	M-1 16.6.76	30 25 36 9 2.0 Mg(ClO <sub>4</sub> ) <sub>2</sub>	1.1 15.3 100 82 33 500	53.9	584 20.2 10.4	357 18.1 45.0	321 18.8 43.0	300 18.8 49.0
8	M-2 16.6.76	do	1.3 9.2 100 85 33 730	33.5	84.6 10.1 36	521 17.7 53.2	468 17.7 41.6	438 18.2 51.0

Table 2 (contd)

1	2	3	4	5	6	7	8	9
9	M-3 16.6.76	30 25 36 9 2.0 Mg(ClO <sub>4</sub> ) <sub>2</sub>	2.08 6.0 90 81 33 770	33.7	892 17.8 19.6	544 17.6 52.3	495 18.7 56.2	462 18.5 55.7

\* Grams of salt per 150 gm solution. Pressure treated at 300 psi; feed concentration = 0.5% NaCl  
Operating pressure = 250 psig.

$t_E$ : Evaporation time (secs);  $V_C$ : speed of fall of casting tube (ft/min);  $T_{HT}$ : Temperature of hot water for annealing membrane (°C)  
 $F_{N_2}$ : Number of bubbles of air circulated/minute during casting,  $T_{RM}$ : room temperature (°C); PWP: pure water permeability at 25°C;  $F_{FR}$ : Brine feed rate through the membrane tube (cc/min),  $N_{Re}$ : Reynolds number as defined in the text; J: water flux at 25°C;  $\%R$ : percentage salt rejection; NONE: No annulus; TPI: Annulus with O-ring turbulence promoter, TP2: annulus with helical turbulence promoter; TP3: annulus only.

are poorer than those with TP2, it may be assumed that the 'O' ring turbulence promoters are quite useful. Further the fact that membranes with TP1 perform better or equal to those with TP3 implies that ring turbulence promoters do have some effect even though TP3 arrangement has large velocity gradient. No investigations were made by varying the 'O' ring gaps to optimize the 'O' ring placement.

Increasing the feed flow rate from 500 cc/minute to 800 cc/minute improves the performance in any arrangement only marginally. The marginal reduction in membrane performance when the flow rate is increased from 500 cc/minute to 800 cc/minute when no annulus is present is somewhat contradictory. On opening the test cell after 800 cc/minute run, it was found that a red deposit has formed in the membrane because of accidental contamination of the feed with some rust. At these low flow rates, dirt if present will accumulate on the membrane surface and performance will deteriorate somewhat. One is, however, likely to see drastic improvement in performance in arrangement (a) (none) only if the tube Reynolds number is increased to very high values of 10000-30000 [1]. For the annuli, similar conditions hold except no experimental data is available in literature on what should be an upper Reynolds number for obtaining the fullest membrane potential.

C. Local Membrane Performance along Tube Length: The axial variation in performance of all tubular membranes studied in

this work was determined by measuring the water flux and salt rejection in seven different locations along the membrane tube. These locations cover the permeate from the whole membrane tube. The results are given in Table 3 and are plotted as a function of the location of the collection tubes in Figures 8 and 9. It may be seen from these figures as well as Table 3 that with no annulus (i.e. None condition of operation), the membrane at location 7 ( $C_7$ ) performs the best, followed by a gradually decrease in membrane performance along the flow direction and then a rise in membrane performance at the last station  $C_1$  before the test cell length is covered. This may be easily explained. At station  $C_7$ , due to entrance conditions, mixing is much better even though  $N_{Re}$  is low so that the membrane performance is good. As the flow goes along the straight tubular membrane, concentration polarization increases along tube length so that membrane performance decreases along tube length. At the very end (location  $C_1$ ) since the flow is leaving the test cell, mixing conditions are good and the membrane performance improves. Performance at  $C_1$  is lower than that at  $C_7$  since the average salt concentration is higher at  $C_1$  and the mixing conditions are never such that the effect of the earlier concentration polarization can be eliminated altogether. These results suggest that with various turbulence promoters put on an annulus introduced in the test cell, membrane performance will be better. Table 3



TABLE 3

## EFFECT OF CONCENTRATION POLARIZATION ON TUBULAR MEMBRANE PERFORMANCE

FOR 0.5 WEIGHT PER CENT NaCl

Membrane* Code No. F <sub>FR</sub>	Type of annulus used (N <sub>Re</sub> )	C <sub>1</sub> J %R	C <sub>2</sub> J %R	C <sub>3</sub> J %R	C <sub>4</sub> J %R	C <sub>5</sub> J %R	C <sub>6</sub> J %R	C <sub>7</sub> J %R	Average J %R
1	2	3	4	5	6	7	8	9	10
M-7 4.6.76 F <sub>FR</sub> =570	None (664)	12.8 38.4	12.7 38.4	14.1 39.6	8.4 36.7	16.6 38.4	11.1 45.5	11.3 60.5	11.7 42.7
do	TP1 (407)	16.2 65.2	14.3 67.5	16.0 68.0	13.3 66.0	14.4 66.0	15.0 68.0	14.4 71.3	14.9 68.0
do	TP2 (364)	16.2 60.0	15.0 62.5	17.6 61.0	11.8 61.4	14.7 62.5	15.0 64.7	14.5 69.0	15.0 63.0
do	TP3 (360)	18.2 60.0	17.1 63.5	17.1 62.6	13.5 63.0	15.8 63.5	15.8 65.0	15.3 68.0	16.6 63.7
M-7 4.6.76 F <sub>FR</sub> =800	None (904)	13.0 40.0	11.5 38.4	12.2 39.2	6.2 39.2	10.8 43.8	10.3 50.8	10.3 62.8	10.9 45.0
do	TP1 (570)	17.6 64.0	17.8 68.0	18.8 67.4	10.2 66.0	15.8 66.4	14.7 69.8	15.8 71.3	15.6 67.5
do	TP2 (515)	16.1 64.2	15.3 65.8	19.3 66.4	18.4 65.8	14.9 65.8	15.3 67.0	14.4 69.0	15.0 66.2
do	TP3 (480)	18.0 64.0	18.2 66.0	19.1 68.4	13.5 68.0	15.6 68.0	17.1 67.0	15.9 71.0	17.3 67.0

Table 3 (contd)

1	2	3	4	5	6	7	8	9	10
M-8 4.6.76	None (589)	14.5 18.8	14.5 19.4	17.7 17.9	15.4 12.1	21.5 11.6	16.6 24.6	14.0 46.4	16.0 21.2
F <sub>FR</sub> =500	TP1 (357)	12.4 52.5	12.8 51.1	13.0 50.7	8.4 47.1	11.8 45.8	12.3 49.7	16.0 56.5	12.0 51.0
do	TP2 (321)	13.7 48.8	10.9 51.1	11.6 53.1	17.5 50.1	10.7 50.1	13.7 53.6	12.4 58.0	12.8 52.1
do	TP3 (300)	16.0 41.5	15.7 42.5	17.7 44.5	15.1 45.5	15.2 44.4	15.9 47.3	15.4 53.1	15.6 48.4
M-8 4.6.76 750	None (870)	11.4 29.0	11.3 27.0	16.4 27.0	10.8 21.8	14.0 21.8	14.0 32.4	11.5 49.6	13.4 30.5
do	TP1 (556)	14.3 54.5	14.2 55.5	15.3 54.0	10.5 53.0	12.3 51.5	15.1 55.5	13.2 60.4	13.4 55.3
do	TP2 (461)	15.4 39.0	12.4 41.5	15.0 38.6	10.5 41.0	16.1 40.6	16.7 40.6	15.7 45.0	16.6 40.8
do	TP3 (432)	17.4 43.0	19.5 46.0	15.9 48.0	11.4 48.0	16.1 48.2	16.9 41.2	16.0 53.6	16.6 48.0

\*For casting conditions and compositions, please refer to Table 2

1. J<sub>w</sub> water flux in U.S. Gallons/ft<sup>2</sup> day

2. R-% salt rejection

3. C<sub>1</sub>, C<sub>2</sub>, ..., C<sub>7</sub> are compartments from the brine out let of the test cell i.e. C<sub>7</sub> is feed entering point

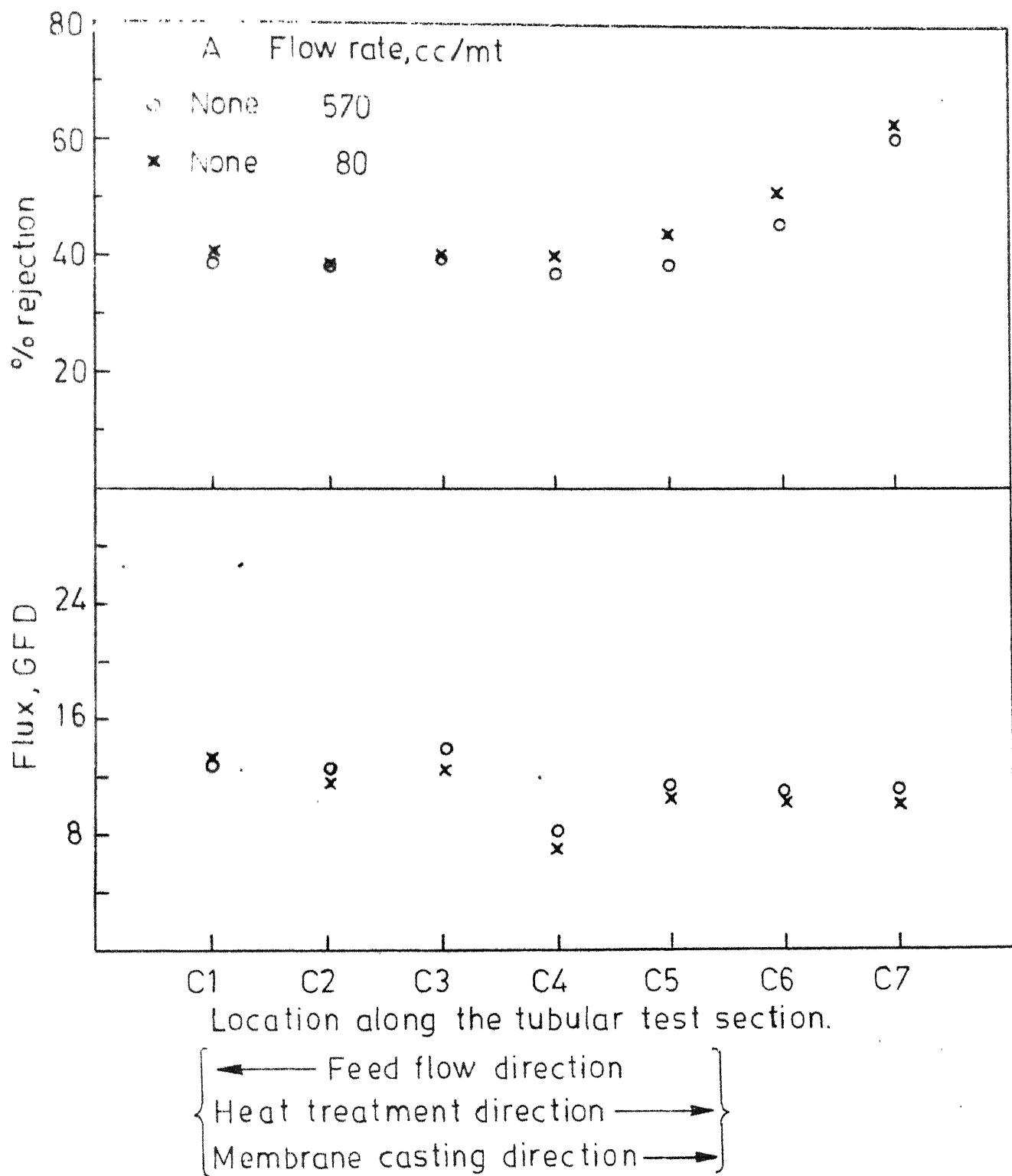


Fig 8 -Membrane performance along the membrane tube length.

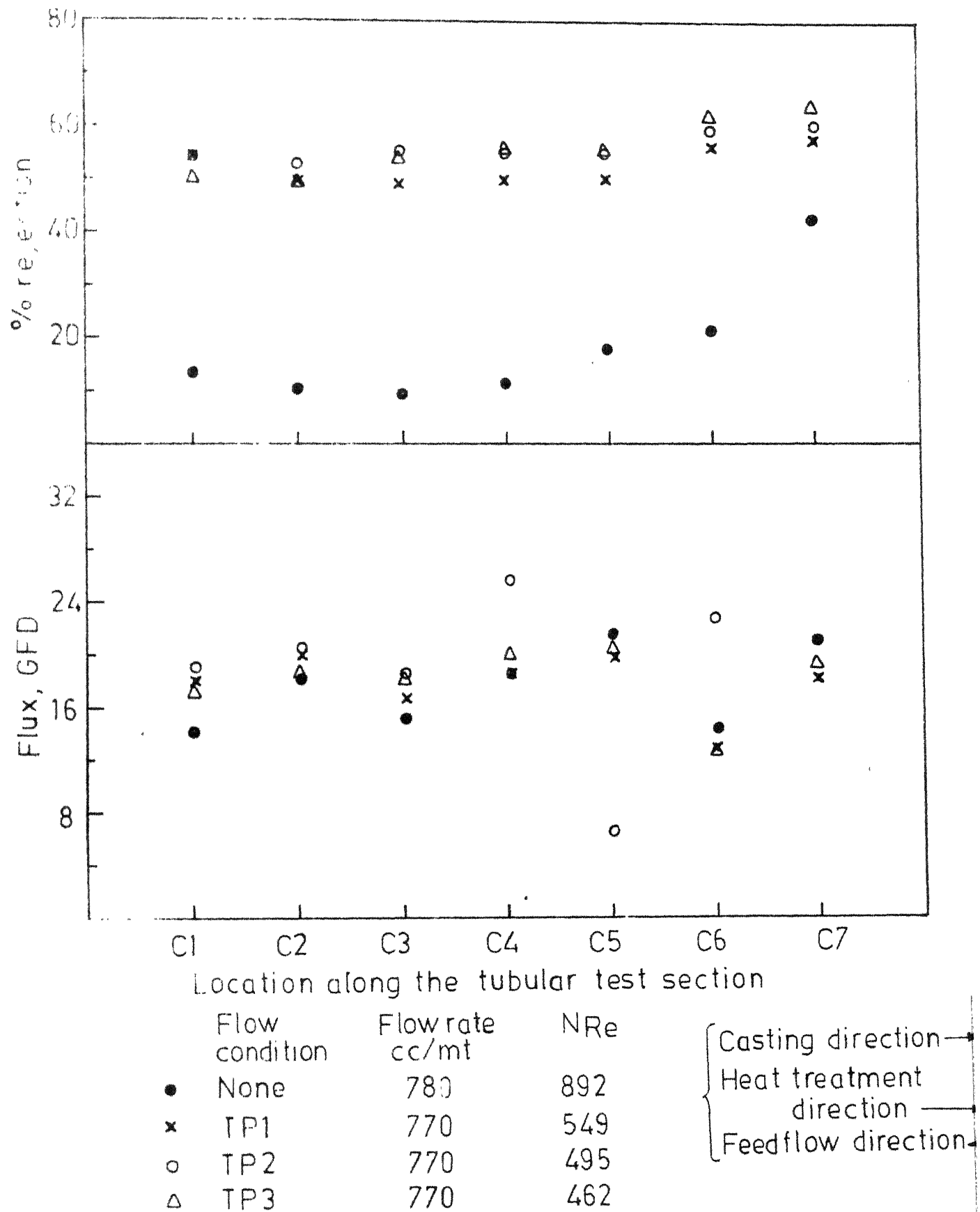


Fig. 9 - Effect of annulus - turbulence promoter on membran performance.

TABLE 4

EFFECT OF AIR BLOWING ON MEMBRANE PERFORMANCE

S. No.	Membrane Code No.	Composition of Casting solution CA = F AC = D **S	Casting Conditions			Testing Conditions			Tubular (TP1)			*Flat		
			t <sub>E</sub> Sec	V <sub>C</sub> ft/min	F <sub>H2</sub> bbl/nt	T <sub>HT</sub> °C	T <sub>RT</sub> °C	F <sub>FR</sub> cc/min	PWP	J	%R	PWP	J	%R
1	2	3	4						6			7		
1	M-2 (9.7.76)	32.0-22.0	0.48			81			49.0			52.0		
		27.6-18.4	20.8			30			30.0			35.5		
		2.44, Mg(ClO <sub>4</sub> ) <sub>2</sub>	<u>NO AIR</u>			680			34.0			52.5		
2	M-3 (9.7.76)	Co	0.37			82			86.5			8.0		
			32.8			33			48.5			45.0		
			<u>40.</u>			600			13.6			54.0		
3	M-5 (16.6.76)	30.0-25.0	0.81			87			60.5			-		
		27.0-18.0	15.3			33			39.0			-		
		2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	<u>20.</u>			600			29.0			-		
4	M-7 (4.6.76)	32.0-22.0	0.95			81			29.0			59.0		
		27.6-18.4	9.2			35			15.6			23.4		
		2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	<u>60</u>			800			67.5			67.5		

\*Results of tubular membrane tested in flat form on flat test cells      \*\* Salt in gm/150 gm solution

The above membranes are pressure treated at 300 psig

Feed concentration = 0.5 weight per cent NaCl, operating pressure = 250 psig.

membranes prepared are as good as the flat membranes already tested by Ghosh [23]. The performances of flat membrane pieces obtained from membrane tubes are given in Table 5. Some of the flat pieces were obtained from untested membrane tubes and were separately heat treated whereas others were cut out from a tested membrane tube taken out of the test cell 1 or 2. It is clearly seen that the flat membrane pieces perform very much better than the tubular membrane lending credence to the presence of extreme concentration polarization in the tubular test cell 1 or 2 under the given low flow rates. Further, a comparison with Ghosh's data indicates that these flat pieces from tubular membranes perform as well as the best membranes cast by him in the flat form. Any apparent discrepancy between the performance of a flat piece and the corresponding tubular membrane is to be traced to the fact that the flat piece may have been taken from an untested section of the tube and it is quite possible to have some amount of performance variation along the membrane tube length due to any uncontrolled variation in casting conditions.

(F) Concentration Polarization as a Function of Membrane Tube Reynolds Number:

One may use Brian's [2] numerical solution to get an estimate of the extent of concentration polarization as a function of tube Reynolds number. Instead of this procedure, we will here quote the experimental measurements of Richardson

TABLE 5

## PERFORMANCE OF TUBULAR MEMBRANES TESTED ON FLAT TEST CELLS

S. No.	Membrane Code No.	Composition of Casting Solution CA = F AC = D * Salt	Casting Conditions		Testing Conditions		J <sub>flat</sub> GFD (J <sub>tube</sub> ) GFD	R <sub>flat</sub> (R <sub>tube</sub> )	Remarks
			t <sub>E</sub> FN2 T <sub>HT</sub>	Sec. ft/min. °C	T <sub>RT</sub> °C cc/min. GFD	F <sub>FR</sub> PWP			
1	2	3		4	5		6	7	8
1.	M-3 25.2.76	32 - 22 46 - 00	1.3 0 72		25 600 22.5		14.7 (7.5)	86.5 (62.5)	This portion was tested before in tubular Test Cell 1
2.	M-1 (8.4.76)	30 - 25 27 - 18	2.5 60 82		25 600 39.0		33.7 -	80.0 -	This was heat-treated in flat form from an untested tube
3.	M-2 (8.4.76)	30 - 20 50 - 00	3.3 60 81		25 600 69.0		54 -	43 -	do
4.	M-2 (8.4.76)	30 - 20 50 - 00 2.44, Mg(ClO <sub>4</sub> ) <sub>2</sub>	1.7 80 81		34.5 600 60.0		42.0 (16.4)	49.5 (34.8)	do
5.	M-2 (14.5.76)	32 - 22 27.6 - 18.4	1.05 80 81		34.5 600 103		58.5 -	53.0 -	do
6.	M-7 (4.6.76)	32 - 22 27.6 - 18.4 2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	0.95 60 81		34.5 600 59		27.8 (15.0)	67.3 (68.0)	do

Table 5 (contd)

1.	2	3	4	5	6	7	8
7.	M-1	30 - 25	1.1	34	34.5	53.5	This portion was tested before in tubular test cell-2
	(16.6.76)	35 - 9	.100	600	(18.1)	(45.0)	
		2.0, Mg(ClO <sub>4</sub> ) <sub>2</sub>	82	94.3			

.\* Salt in gms/150 gm solution

All membranes were pressure treated at 300 psig and operated at 250 psig for 0.5% NaCl feed solution.



et al. [26]. They had operated a 61 cm long, 2.26 cm diameter CA tubular membrane at 800 psig pressure with a 1.015 weight per cent NaCl feed solution in the Reynolds number range of 429 to 19284. Those results of their experiment that are of primary interest to us are the following: (a) At a Reynolds number of 429, the averaged membrane flux was 15.0 GFD and the overall per cent salt rejection was 81 per cent corresponding to a salt reduction factor of 6.01 (defined as feed concentration/permeate concentration). (b) On the other hand, at a Reynolds number of 19,284, the averaged membrane flux was 23.3 GFD and the per cent salt rejection had gone up to 96 per cent corresponding to a salt reduction factor of 21.7. (c) Further, at a Reynolds number of 1,286, the averaged membrane flux had risen only marginally to 16.6 GFD from that of 15.0 GFD for  $N_{Re} = 429$  and similarly the salt reduction factor had gone upto only 6.37 from 6.01 obtained at  $N_{Re} = 429$ . A salt reduction factor of 6.37 corresponds to a per cent salt rejection of 83.5 per cent.

The above results indicate the severity of concentration polarization at low Reynolds number. For this reason, little purpose is served by comparing the performance of the tubular CA membranes of the present work with those of Sourirajan et al. [10] who happen to be the only other investigators who have operated tubular reverse osmosis membranes at 250 psig.

However, comparison of flat membrane performances at 600 psig by Pandurangaiah [14] has already confirmed the superiority of the present type of membranes over those of Sourirajan. At the lower pressure of 250 psig, this difference is likely to be increased as the investigations of Ghosh [23] suggest.

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## CHAPTER 4

### CONCLUSIONS

Completely defect free CA tubular membranes have been cast, with good control of casting parameters for low evaporation times. These membranes can be heatreated almost uniformly without damaging the membrane skin structure along the whole membrane tube length even at the ends where seals are made. Low performance characteristics of the tubular membranes obtained at 250 psig with 0.5 weight per cent NaCl feed were only due to severe concentration polarization of salt over the membrane surface, resulting from very low feed flow rate corresponding to low Reynold's numbers. Characterization of tubular membranes and studying the effect of turbulence promoters in laminar flow of low Reynolds number ( $N_{Re} \quad 2000$ ) have limited utility. Therefore a pump of very high capacity is absolutely essential for adequate membrane characterization and optimization of turbulence promoter arrangements vis-a-vis a given Reynolds number.     abric

The fabrication details and the experiments carried out enable us to conclude confidently that low pressure tubular reverse osmosis (as developed in our laboratory) is to be pursued with greater vigor for immediate commercialization.

## CHAPTER 5

### SUGGESTIONS FOR FURTHER WORK

On the basis of the present work, the following investigations should be undertaken in order to make the present developments a total success:

(i) Study more extensively the various casting variables such as air-flow rate, the composition of gelation bath and the range of evaporation period in the case <sup>of</sup> tubular membranes cast in the present casting equipment.

(ii) Develop suitable membranes for sugar solution concentration, separation of glycerine from spent liquid from soap industry.

(iii) Investigate the possibility of heat treatment of tubular membranes in the unrestrained form.

(iv) Place teflon caps at the ends of the annulus to facilitate easy insertion into the membrane tube.

(v) Change end seal tube material to Nylon to avoid possible damages to the membrane ends during mounting and for easy assembling.

(vi) Change the bob hanging arrangement for better centring with casting tube. It should be such that bob position could be changed to any required position in x-y-z directions.

(vii) Study the turbulence promoter effect for optimal parameters of turbulence promoter such as shape, sizes,

spacing for tubular membranes and feed systems.

(viii) Using a high pressure pump of large capacity, true performance characteristics of tubular membranes should be evaluated.

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APPENDIX1. Conversion Factor Calculations for converting 100 cc/hr. permeate to GFD units (U.S. gallons per day per sq. foot):

(A) Test Cell L: Length of the membrane under each compartment (1) =  $\frac{22}{6}$  inch.

I.D. of tubular membrane = 0.8 inch.

∴ Membrane area for water permeation

per compartment =  $\pi D_1 l = \pi \times 0.8 \times \frac{22}{6}$

$$= 9.22 \text{ inch}^2$$

$$100 \text{ cc per hour} = 100 \text{ cc} \times \frac{0.001 \text{ lit.}}{\text{cc}} \times 0.2642 \frac{\text{gallons}}{\text{litre}}$$

$$\times \frac{1}{\text{hr}} \times \frac{24 \text{ hr}}{\text{day}} = 0.635 \frac{\text{gallons}}{\text{day}}$$

∴ 100 cc per hour per 9.22 in.<sup>2</sup> area

$$= 0.635 \frac{\text{gallons}}{\text{day}} \times \frac{1}{9.22 \text{ in.}^2} \times \frac{144 \text{ in.}^2}{\text{ft}^2}$$

$$= 10.00 \frac{\text{gallons}}{\text{ft}^2 \text{ day}}$$

100 cc/hr/compartment of the membrane = 10.0 GFD (US GFD)

(B) Test Cell 2:

(a) For compartments 1 and 7

Membrane length = 3.5 in.

Membrane area =  $\pi \times 0.8 \times 3.5 = 8.79 \text{ in.}^2$

$$\begin{aligned} \therefore 100 \text{ cc/hr}/8.79 \text{ in}^2 \text{ area} &= 0.635 \times \frac{1}{8.79} \times 14.4 \\ &= 10.46 \text{ GFD} \end{aligned}$$

(b) For Compartments 2,3,4,5 and 6:

Membrane length = 3.0 in.

Membrane area =  $\pi \times 0.8 \times 3.0 = 7.54 \text{ in.}^2$

$$\therefore 100 \text{ cc/hr}/7.54 \text{ in.}^2 \text{ area} = 0.635 \times \frac{1}{7.54} \times 144 \\ = 12.1 \text{ GFD}$$

## 2. Reynolds Number Calculations for Various Flow Configurations:

$$N_{Re} = \frac{\rho v D_e}{\mu} \quad (1)$$

where  $\rho$ ,  $v$ ,  $\mu$ , are density, average velocity, viscosity of the feed and  $D_e$  is equivalent diameter of the flow system.

$$\text{For annulus } D_e = D - d \quad (2)$$

where  $D$  is inside diameter of outside tube and  $d$  outside diameter of inside tube making the annulus.

$$v = \frac{Q \times 4}{60 \times \pi (D^2 - d^2)} \text{ cm/sec} \quad (3)$$

where  $Q$  is volumetric flow rate cc/minute.

Substituting value of  $D_e$  and  $v$  in (1) gives

$$N_{Re} = \frac{\rho}{\mu} \times \frac{Q \times 4}{60 \pi (D^2 - d^2)} \times (D - d) \\ = \frac{Q}{\mu 15\pi (D + d)} \quad (4)$$

(a) None Condition:

Here,  $d = 0.0$ ,  $D = 0.8 \text{ in.} = 2.03 \text{ cm}$ ,  $\rho = 1 \text{ gm/cc}$

$\mu = 0.90 \text{ cp}$

$$\therefore N_{Re} = \frac{Q \times 1}{15\pi \times 0.90 \times 10^{-2} \times 2.03} = \frac{Q}{0.86}$$

e.g.  $Q = 500$  cc/minute

$$N_{Re} = \frac{500}{0.86} = 581$$

(b) TP1

Here,  $d=0.5$  in. = 1.27 cm.,  $\rho = 1$  gm/cc,  $\mu = 0.9$  cp

$$N_{Re} = \frac{Q \times 1}{15\pi \times 0.9 \times 10^{-2}} (2.03 + 1.27)$$

$$= 0.716 Q$$

e.g.  $Q = 500$  cc/minute

$$N_{Re} = 358$$

(c) TP2

In this case,  $d=0.65$  in. = 1.65 cm,  $\rho = 1$  gm/cc,  $\mu = 0.9$  cp

$$N_{Re} = \frac{Q \times 1}{15 \pi \times 0.9 \times 10^{-2}} (2.03 + 1.65)$$

$$= 0.641 Q$$

e.g.  $Q = 500$  cc/minute,

$$N_{Re} = 0.641 \times 500 = 321$$

(d) TP3

Here,  $d=0.75$  in. = 1.9 cm,  $\rho = 1$  gm/cc,  $\mu = 0.9$  cp

$$N_{Re} = \frac{Q \times 1}{15 \pi \times 0.9 \times 10^{-2}} (1.9 + 2.03)$$

$$= 0.6 Q$$

e.g.  $Q = 500$  cc/minute

$$N_{Re} = 0.6 \times 500 = 300$$

### 3. Sample Calculations:

The following sample calculations given separately for individual compartments and for the overall test cell are for membrane tube M-7 (4-6-76) tested in test cell 2 and NONE flow condition at a feed rate of 570 cc/minute

#### (a) Individual Compartment Calculations:

##### Pressure Treatment Step:

At compartment 1 ( $C_1$ ),

Volume of permeate collected = 84 cc

Time period of collection = 20 minutes

Conversion factor for this compartment = 10.4 GFD/100 cc/hr

$$\begin{aligned} \text{Pure water permeability (PWP)} &= \frac{84 \times 60}{20 \times 100} \times 10.4 \\ &= 26.2 \text{ GFD} \left( \frac{\text{US Gallons}}{\text{ft}^2 - \text{day}} \right) \end{aligned}$$

Average temperature of the feed = 35°C

Viscosity of water at 25°C = 0.954 cp

Viscosity of water at 35°C = 0.800 cp

$$\text{Permeate flux at 25°C} = 26.2 \times \frac{0.800}{0.954} = 21.2 \text{ GFD}$$

##### Desalination Run:

Again at  $C_1$ ,

Volume of permeate collected = 48.5 cc

Time period of collection = 20 minutes

Conversion factor for this compartment = 10.4 GFD/100 cc/hr

Average temperature of the feed = 35°C

$$\text{Permeate flux} = \frac{48.5}{20} \times 60 \times \frac{10.4}{100} = 15.1 \text{ GFD}$$

Viscosity of water at  $35^{\circ}\text{C} = 0.8 \text{ cp}$

$$\text{Permeate at } 25^{\circ}\text{C} = 15.1 \times \frac{0.8}{0.954} = 12.8 \text{ GFD}$$

Salt Rejection:

Volume of permeate solution used for titration = 1.0 cc

Volume of  $\text{AgNO}_3$  solution (about 0.009N) required  
= 5.30 cc

Volume of  $\text{AgNO}_3$  solution when it was titrated against  
1.0cc of feed solution = 8.60

$$\text{Per cent rejection} = \frac{8.60 - 5.30}{8.60} \times 100 = 38.4 \text{ per cent}$$

Since per cent salt rejection is defined as

$$\text{Per cent R} = 100 \times \frac{\text{Salt concentration in feed} - \text{salt concentration in permeate}}{\text{Salt concentration in feed}}$$

(b) Average Values Over Entire Test Cell:

Pressure Treatment Step: Permeate collected per 20 minutes at

$C_1, C_2, C_3, C_4, C_5, C_6, C_7$  are 84, 73.5, 79, 41.5, 61, 78 and 81.5 cc, respectively.

$$\begin{aligned} \text{Total permeate collected over entire test cell} &= 84 + 79 + 41.5 \\ &+ 73.5 + 61 + 78 + 81.5 = 498.5 \text{ cc} \end{aligned}$$

Time period of collection = 20 minutes

$$\text{Total membrane area} = \pi D l = \pi \times 0.8 \times 22 = 55.2 \text{ in}^2$$

$$\text{Average pure water permeability} = \frac{498.5 \text{ cc}}{20 \text{ min.}} \times 0.001 \frac{\text{litre}}{\text{cc}}$$

$$\begin{aligned} &\times 0.2642 \frac{\text{gallon}}{\text{litre}} \times \frac{60 \text{ min.}}{\text{hr}} \times \frac{24 \text{ hr}}{\text{day}} \times \frac{144}{55.2} \\ &= 24.8 \text{ GFD} \end{aligned}$$

Average feed temperature =  $35^{\circ}\text{C}$

Viscosity of water at  $35^{\circ}\text{C}$  = 0.8 cp

$$\begin{aligned}\text{Average pure water permeability at } 25^{\circ}\text{C} &= \frac{24.8 \times 0.8}{0.954} \\ &= 22.2 \text{ GFD}\end{aligned}$$

Desalination Step:

Permeate collected at  $C_1, C_2, C_3, C_4, C_5, C_6$ , and  $C_7$  per twenty minutes are 48.5, 41.5, 46.0, 27.5, 38.0, 36.0 and 43.0, respectively.

Total permeate collected over the test cell = 280.5 cc

Time period of collection = 20 minutes

$$\begin{aligned}\text{Average permeate flux} &= \frac{280.5}{20} \times 0.001 \times 0.2642 \times 60 \times 24 \\ &\times \frac{144}{55.2} = 13.9 \text{ GFD}\end{aligned}$$

$$\begin{aligned}\text{Average permeate flux at } 25^{\circ}\text{C} &= \frac{13.9 \times 0.80}{0.95} \\ &= 11.73 \text{ GFD}\end{aligned}$$

Overall Percentage Salt Rejection:

Here 1 cc permeate of each  $C_1, C_2, C_3, C_4, C_5, C_6$  and  $C_7$  respectively require 5.3, 5.3, 5.2, 5.45, 5.3, 4.7 and 3.4 cc of  $\text{AgNO}_3$  solution ( 0.009 N)

Volume of  $\text{AgNO}_3$  required when it was titrated against 1 cc of feed solution = 8.6 cc

$$\begin{aligned}\therefore \text{ per cent rejection} &= 5.3 \times 48.5 + 5.3 \times 41.5 + 5.2 \times 46.0 \\ &\quad + 5.45 \times 27.5 + 5.3 \times 38.0 + \dots \\ &\quad + 4.1 \times 36.0 + 3.4 \times 43.0 \\ &= \frac{8.6(48.15 + 41.5 + 46.0 + 27.5 + 38.0 + 36.0 + 43.0)}{100} \\ &= 42.67 \text{ per cent}\end{aligned}$$

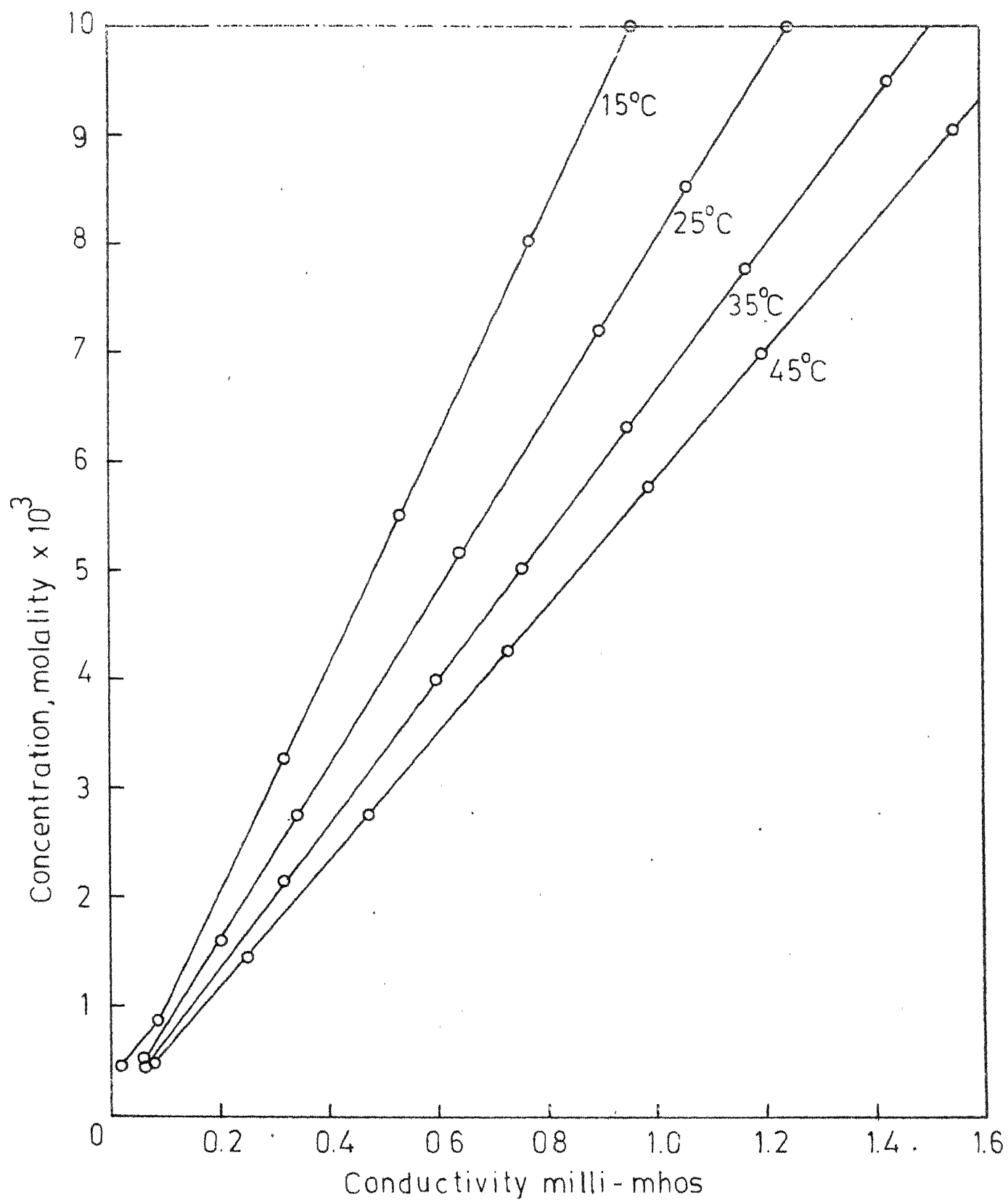


Fig. 10 - Conductivity of sodium chloride at different temperature and at different concentrations.